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VOLUME II CONTAMINANT GENERATION AND SENSITIVITY STUDIES

FINAL REPORT
SHUTTLE
FILTER
STUDY

WINTEC

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FINAL REPORT

SHUTTLE

FILTER

STUDY

Approved

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SECTION A

CONTAMINANT GENERATION STUDIES AT THE COMPONENT LEVEL

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FOREWORD

Volume II of the "Shuttle Filter Study" Final Report consists of two major sections. The section entitled, "Contaminant Generation Studies at the Component Level," describes unique test methods to determine the degree of contamination released by typical operating components. Several different components were tested and the results of these tests are described.

The section entitled, "Component Sensitivity Test," describes a method to determine the level of particulate contamination at which an operating component will fail to develop its design life. The degree of protective filtration required may be determined from the test results. Tests conducted on a typical operating component, a bi-propellant valve, are described in detail.

ABSTRACT

Contaminant Generation Studies were conducted at the component level using two different methods, radioactive tracer technique and gravimetric analysis test procedure. Both of these were reduced to practice during this program. In the first of these methods, radioactively tagged components typical of those used in spacecraft were studied to determine their contaminant generation characteristics under simulated operating conditions. Because the purpose of the work was 1) to determine the types and quantities of contaminants generated and 2) to evaluate improved monitoring and detection schemes, no attempt was made to evaluate or qualify specific components. The components used in this test program were therefore not flight hardware items. Some of them had been used in previous tests; some were obsolete; one was an experimental device. They were supplied for the purpose of these tests by NASA and NASA contractors.

In addition to the component tests, various materials of interest to contaminant and filtration studies were irradiated and evaluated for use as autotracer materials. These included test dusts, plastics, valve seat materials, and bearing cage materials.

In all, five components were tested. These included:

- Two types of solenoid valves,
- An augmented spark igniter valve assembly,
- A hydraulic actuator,
- A bearing test device.

These components were selected because they were representative of the basic motions involved in wear, i.e., sliding surfaces, rotating motion, impact, etc.

The test procedure involved operating the component a predetermined number of cycles in a test loop. A circulating fluid removed contaminants for collection on filter screens. Either water, hydraulic fluid, or liquid nitrogen was used. Nuclear detection techniques were used to determine the type and quantity of contaminants collected on the filters.

With these techniques, it was possible to make on-line measurements of contaminant generation. In most cases the source of the contaminant could be identified, i.e., "valve body," "bearing race," etc. Sensitivity was quite good using these methods, since test results indicated that 10 micrograms of typical contaminant materials can be detected with ease. Using more sophisticated techniques, the limit of detectability can be extended to 1 nanogram, but there seems to be no practical incentive to do so for the purposes of these tests.

1.0 CONTAMINANT GENERATION PARAMETERS

1.1 Definition of Main Parameters

Parameters that affect the performance of typical fluid system components can be divided into three categories for this study. They are:

- Source of Contaminants
  - contaminants initially in the system
  - contaminants external to the system that enter when it is opened or loaded
  - system generated contaminants
- Effect of Contaminants on Components
- Performance Degradation or Failure Due to Contaminant Effects

For convenience, in the discussion that follows, these three categories of parameters are referred to as SOURCE parameters, EFFECT parameters, and PERFORMANCE parameters.

1.2 SOURCE Parameters

We assume that suitable design of ground facilities to minimize external contamination is technically feasible. Therefore, it is excluded from further discussion.

Contaminants initially in the system can come from a variety of sources. Table I lists typical types of internal contaminants as well as parts of a fluid system where they can be found.

System generated contaminants can arise from the operation of various components with moving surfaces. These particles are formed by mechanical action when surfaces mutually fracture and splinter into each other, or by chemical action (corrosion), or by vibration or impact. Figure 1 shows potential sources of system generated contaminants.

1.3 EFFECT Parameters

Contaminants can affect moving mechanisms, clog filters, cause flow erosion, and lead to deterioration or modification of fluid parameters. The most serious of these effects are those which are related to moving mechanisms. Figure 2 summarizes the typical effects.

1.4 PERFORMANCE Parameters

When the operation or behavior of components is modified by contaminants, the result is altered performance. The performance parameters affected will determine the sensitivity of the component to contamination. For some parameters, sensitivity will be

TABLE 1: TYPES AND SOURCES OF CONTAMINATION IN A TYPICAL FLUID SYSTEM

COMPONENTS (SOURCE)	CONTAMINANTS								
	Oxide Scale	Plastics & Elastomers	Fluid Additives	Metal Particles	Air-borne Dirt	Silica Sand	Lapping Compound	Process Residues	Fibers
Fluid Medium	1		1		1	1			1
Tank	1	1		1	2	1	1		1
Relief Valve		1		2	1	1			1
Accumulator (bladder & piston types)	1	1		1	1			1	1
Filter	1	1		1	2				1
Piping, fittings, etc.	1	1		1	2			1	1
Control Valves		1		1	1		1		1
Actuators		1		1	1	1			1
Pump		1		3	1				1

.1 = noticeable;   2 = medium;   3 = strong

Reference: Howell, Fluid Components Handbook, 1964, Table 10.2.2.1

FIGURE 1: SYSTEM GENERATED CONTAMINANT SOURCES
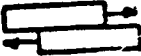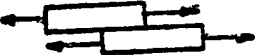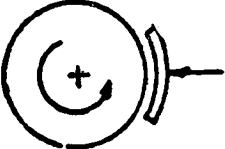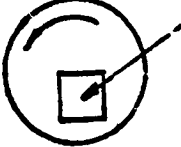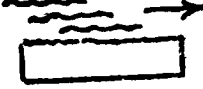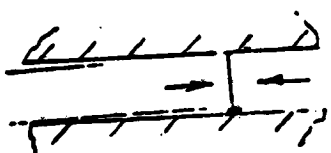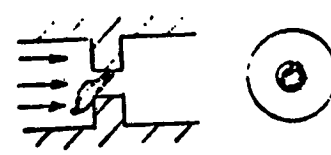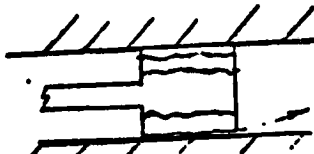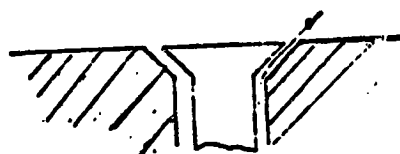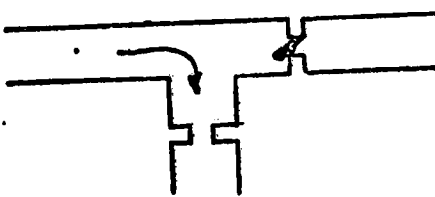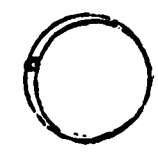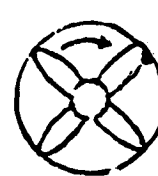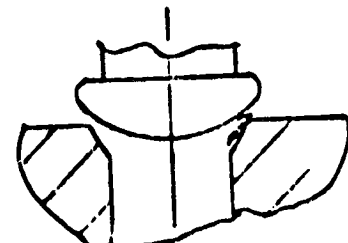
	DESCRIPTION	EXAMPLE
a.	Rectilinear motion (one-directional)	Fluid flow, feed screw
b.	Rectilinear motion (periodic)	Piston and cylinder
c.	Rotational (one-direction) Radial force	Radial pump housing, bushings
d.	Rotational (one-direction) Axial force	Axial pump housing
e.	Rolling	Bearing
f.	Impact	Solenoid, actuators
g.	Vibration	Pipe flexure, turbine vibration
h.	Surface conditions	Corrosion or surface finish

FIGURE 2:   EFFECTS OF CONTAMINANTS ON TYPICAL COMPONENTS

	DESCRIPTION	EXAMPLE
a.	Sticking of sliding mechanisms	Dirt lock, Stiction, Weldment
b.	Blocked flow passage	Plugged metering orifice
c.	Scored surfaces	Scoring of shafts, rods and slides causing leakage paths.
d.	Wear and friction	Abrasion of seals, increased friction through dirt lock
e.	Altered flow directions (unbalanced system pressure)	Dammed lines or orifices
f.	Misalignment	Misalignment of spools, slide gates, etc.
g.	Jamming of mechanisms	Jamming of impeller type meter flow
h.	Contact Prevention (incomplete closure)	Valve leakage

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much greater than for others. Typical parameters include:

- Leakage rate
- Torque, force, and breakout force requirements
- Pressure decay
- Excessive pressure drop
- Flow decay
- Flow restriction or modification
- Overheating due to friction
- Reproducibility of operation
- Increased power consumption
- Material integrity
- Structural integrity
- Degradation of another component
- Dimensional tolerance or misalignment

In conclusion, it can be seen that numerous paramters are required to specify the performance of the components in a system. Each component involves some but not necessarily all of the test parameters. The cost of testing and qualifying every component would be prohibitive, since the number of parameters and their relationship are quite complex, even for seemingly simple components.

Therefore, typical components were selected in an effort to examine the performance of representative items. Although the components tested by no means include examples of every type found in a spacecraft fluid system, an effort was made to include in the test program examples of each of the sources shown in Figure 1.

2.0 RADIOACTIVE TRACER TECHNIQUES

2.1 Introduction

This section describes radioactive tracer technique for studying the effects of con-
taminants and for studying component contaminant self-generating characteristics in
fluid systems used on spacecraft. The methods utilize radioactive tracers and neutron
activation analysis techniques to achieve a high sensitivity for analysis, to allow
quantification of contaminant concentration and particle size, and to make rapid, re-
mote detection of contaminants possible.

The need for this work arose when it became apparent that conventional gravimetric
methods are limited in application. The main limitations are:

> Sensitivity is limited to 0.1 — 1.0 milligram.

> Difficult to discriminate between contaminants generated by test items
> and other foreign material which might be present in a test loop.

> Remote detection or "on-line" measurement of contaminants is not possible.
> Test system must be perturbed to determine amount of contaminants present.

> In some fluids, such as hydraulic fluids, it is difficult to get accurate
> gravimetric results.

> In all cases, care must be exercised to avoid loss of contaminants before
> weighing.

Using radioactive tracers is a low-cost, convenient way of circumventing each of the
above problems.

2.2 Methods

Two main types of tests were conducted under this program. In one part of the program,
tests were run with "test dusts" or other types of simulated contaminants. These in-
cluded such things as AC-coarse and AC-fine tests dusts (Arizona road dusts), which do
not necessarily represent the type of contaminants found in actual spacecraft systems.
However, they are useful as a device for making comparative studies of various types of
filters, and for evaluating certain wear mechanisms.

When test dusts are used, the objective is usually to evaluate filter performance or
to determine the effect of contaminants on a particular component.

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In a real system, each component must also be considered as a source of contaminant. Thus the problem can be divided into two parts:

(1) Determining effects due to contaminants added to system.

(2) Determining effects due to components generating contaminants.

From this introductory discussion it can be seen that in theory every component such as a valve, bearing, or hydraulic actuator should have two filters: one "upstream" to prevent system contaminants from causing it to malfunction, and another "downstream" to prevent contaminants it generates from affecting other components in the system.

Obviously, the filter requirements for a particular fluid system can not be specified in an optimum way unless the precise performance characteristics of each component are known. These include its propensity to generate contaminants (rate, particle size distribution, amount, etc.) as well as its sensitivity to contaminants in the system from other sources.

One of the objectives of this work was to develop improved methods for studying typical spacecraft components in order to gain information concerning their performance.

Tagged Contaminant Studies

Figure 3 illustrates the method when radioactively tagged test dusts are introduced into a test system.

The sensitivity of selected components to different types of contaminants can be studied in this way. To quantify the analysis and make a rapid, highly sensitive, and remote detection technique possible, the selected contaminants are irradiated to provide low level radioactivity for tracing purposes. Different compounds could be selected to provide distinct isotopes as a tracer for each particle size range of interest. Typical examples are shown in Table 2. This list is not complete; many other types and particle sizes are available and could be used if desired.

Since each isotope has its own characteristic gamma spectrum, it is possible to determine the particle size distribution remotely.

Another advantage of the radioactive tracer technique is that it allows truly quantitative work to be performed. When test dust is added to a test system, some of it lodges on the walls of piping and fittings. Other dust or particles may be unavoidably present, if for no other reason than because the test loop itself generates them. The radioactive method ignores these extraneous sources of contaminants and yet allows a correction for unavoidable losses. If necessary, quantitative mass balances can be made by assaying

TABLE 2

SOME POSSIBLE TAGGED CONTAMINANTS
(Test Dusts)

DESCRIPTION	TAGGING ISOTOPE	PARTICLE SIZE (microns)
Red iron oxide	Fe-59	0.3 - 1.1
Molybdenum	Mo-99	1 - 10
Glass_beads	Na-24	10 - 50

Figure 3

TAGGED CONTAMINANT STUDIES

Contaminant
Test Dusts

irradiate in
nuclear reactor
to low specific
activity

$\phi(t)$
(neutron irradiation)

place
contaminants
in Test Rig

Test
Section

filter

fluid and contaminants

Examine Test Specimen
for Wear

Examine filters
for contaminant
concentration
and types

counting
instrumentation

gamma detector

shield

filter

counts

energy

Characteristic gamma spectra
reveal which contaminants
are collected

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the pipes and fittings until all of the test dust has been acc   t d for.

Component Autotracer Studies

Emphasis in this program has been placed on determining the contaminant generating char-
acteristics of representative spacecraft components. Figure 4 shows a sketch of the
test procedure used for these measurements.

The procedure requires that the component first be examined to determine which of the
sources shown in Figure 1 apply. When this step has been completed, the appropriate
parts of the component are removed and irradiated to provide the specific activity
needed for a test of given duration.

The specific activity to be used depends on the duration of the test, the amount of
contaminant generated, the sensitivity of the detecting instrumentation, the shielding
and health physics limitations, and applicable licenses and safety regulations. In
general an appropriate analysis by a qualified nuclear engineer is required for each
test.

Monitoring by a health physicist during irradiation and testing is necessary to insure
that safety requirements and State and Federal regulations are met. The health physicist
also monitors decontamination of the system, waste disposal, and personnel exposure
records. While the use of radioactivity imposes certain additional complications in the
test procedures, they are by no means restrictive. Comparable measures are taken to
provide an adequate measure of safety when working with most industrial processes
involving cryogenic fluids, corrosive fluids, inflammable materials, and other potentially
hazardous agents. Besides, in general the amounts of radioactivity involved are quite
small, and the risk of personnel exposure or injury is negligible. Care must be exer-
cised principally to insure compliance with the appropriate laws and regulations.

The autotracer method has a number of unique advantages. First, contaminants can be
routinely detected with excellent sensitivity. Consider the following example:

Sensitivity of Method

An irradiated sample of 304 stainless steel, i.e., a bearing or structural part is
allowed to operate in a test system. The generated contaminants are trapped in a filter
or filters. The filters are then counted in a 3" x 3" NaI scintillation counter. The
equation which relates the filter residue count rate to the irradiation and counting
parameters is:

Figure 4

SELF-GENERATED CONTAMINANT STUDY
(Autotracer Technique)

(a) Irradiate wearing surfaces of component in nuclear reactor

$\phi(t)$

Safety studies and experimental design

(b) Place component in test loop under specified operating conditions

Detector and recorder detect wear as a $f(t)$

filter

Fluid Reservoir

test section

pump

(c) Test and examine components for wear

(d) Detect presence of self-generated contaminants in filter using gamma spectroscopy

detector

filter

shield

recorder

counts

energy

(e) Analysis, Calculations, and Repc

$$A = \frac{\eta W \Sigma_a \phi}{\rho} (1 - e^{\lambda \tau}) \, e^{\lambda \tau} \, \alpha \beta \gamma$$

η = 3" x 3" detector efficiency (35%)

W = Weight of contaminant, grams (10^{-6} grams)

Σ_a = macroscopic absorption cross section

ρ = density of contaminant material

λ = decay constant

τ = irradiation time (0.5 hr)

t = elapsed time between end of irradiation and counting

α = % isotope in element

β = % element in 304 S.S.

γ = % γ ray output/disintegration

Two irradiations are considered: 1) a half hour irradiation with thermal neutrons (Table 3) and 2) a half hour irradiation with 14 MeV neutrons (Table 4). Examination of the tables clearly indicates that for both thermal and 14 MeV neutron irradiations, the isotope Manganese-56 is produced in sufficient quantity to allow reliable detection of a 1 microgram quantity of 304 stainless steel deposited on the filter. In addition, the remaining isotope activities are orders of magnitude below the Mn-56 activity, resulting in no interference. The 2.6 hr half-life of Mn-56 is sufficiently long to allow the counting to commence as long as 10 to 20 hours after irradiation while retaining sufficient sensitivity. However, after a few days, the system will have lost the activity due to decay of Mn-56. For example, after 3 days, the Mn-56 residue still within the system will have retained only 5×10^{-8} of its original activity, which is far below any measureable or hazardous quantity.

These calculations were made only for 304 stainless steel, but since all stainless steels contain similar amounts of manganese and iron, these calculations or similar ones could be applied to all stainless steels. A similar approach would be used to evaluate other materials.

Depending on the detection system, irradiation time, and materials used, trace quantities as low as 10^{-9} gram can be detected accurately. Using low level sources, 1 microgram (10^{-6} gram) sensitivities can be attained routinely using this method.

Particle Size Distribution

The particle size distribution of contaminant particles can be estimated by using several filter screens with different mesh sizes. The relative count rates are proportional to

Table 3

THERMAL NEUTRON IRRADIATION OF 304 SS
(one-half hour irradiation)

Isotope	β Composition W/O in S.S.	$t_{1/2}$ Half Life	E_γ Gamma Energy & Yield (Mev(%))	σ_{th} Cross Section (barns)	$\frac{\Sigma_a W}{\rho}$	α Abundance (%)	A Activity* (cps)	Count Rate per μgm (cpm)
C	.08	--	---	--	---	---	---	---
Cr^{50}	18-20	27.8 days	0.32 (9%)	16.0	7.5×10^{-9}	4.3	7.8	15
Ni^{64}	8-12	2.55 hr	1.48, 1.11 (29%)(14%)	1.7	2×10^{-10}	1.16	3.3	20
Si^{30}(max)	1	2.62 hr	1.27 (0.07%)	0.1	6.5×10^{-10}	3.09	11.3	0.16
Mn^{56}(max)	2	2.58 hr	0.84, 1.811 (100%)	13.3	1.1×10^{-8}	100.0	182.0	3800
S (max)	.03	--	---	--	---	---	---	---
P (max)	.045	--	---	--	---	---	---	---
Fe	--	long	low	low	---	---	---	---

*Isotope weight only.

Table 4

14 MeV NEUTRON IRRADIATION OF 304 SS

(one-half hour irradiation)

Isotope	β Composition W/O in S.S.	$t_{1/2}$ Half Life	E_γ Gamma Energy & Yield (Mev(%))	σ_{th} Cross Section (barns)	$\frac{\Sigma_a W}{\rho}$	α Abundance (%)	A Activity* (cps)	Count Rate per μgm (cpm)
$Cr^{52}(n,p)V^{52}$	18-20	3.76 m	---	.07	1	--	---	---
$Ni^{58}(n,2n)Ni^{57}$	8-12	36 hr	.127,1.32,1.89 (14%)(86%)(14%)	.04	3.3×10^{-11}	67.7	0.35	0.73
$Ni^{61}(n,p)Co^{61}$	8-12	99 m	.072 (100%)	.18	8.5×10^{-11}	1.25	0.056	0.12
$Si^{30}(n,\gamma)Si^{31}$	--	--	---	.00049	---	--	---	---
$S^{34}(n,\alpha)Si^{31}$	0.03	2.62 hr	1.26 (.07%)	.14	2.05×10^{-8}	4.27	19.5	0.12

the mass collected on each screen.

Origin of Contaminant Particles

By performing gamma ray spectroscopy, it is possible to determine the isotopes prevalent in each material used in a component. If different materials are used, for example, in a valve body and seat, then the gamma spectra will reveal which material is producing the contaminant particles and how much (relatively) each material generates contaminants. The requirement for different materials is not an absolute limitation to the method, since there are other variables. For example, the same material can sometimes be irradiated for different times or with different particles to emphasize different isotopes. Alternatively, the surfaces of specific parts can be coated or alloyed with materials incorporating trace quantities of appropriate isotopes.

Real Time Measurements

Because the measurement technique uses penetrating gamma radiation, it is not necessary to open the system and to remove the collection devices in order to measure the mass of the contaminants. The measurements can be made remotely (e.g., through a pipe or fitting wall) and continuously, while the component or process is in operation. By selecting the appropriate isotope, autotracers with "shelf-lives" ranging from hours to years can be produced. Thus the test method is applicable to any time span requirement likely to be encountered in component testing.

A number of variations on the basic method are possible, depending on the instrumentation available and specific test requirements. T.P. 1 lists a typical test procedure based on the methods described above.

2.3 Results

System Tests and Calibration

The detector system used one or two channels, depending on the tests being conducted. Each channel consisted of a detector (scintillation detector or GM tube), ratemeter, power supply, and strip chart recorder. In some cases a single channel analyzer (pulse height discriminator) and timer-scalar were used.

The detectors were enclosed in specially fabricated shields and positioned adjacent to flanges containing filter screens. Expedient shielding was placed around the irradiated test component to reduce the background radiation level. The system was initially checked out using an 8 μCi Cs-137 check source. Following this, it was tested by inserting

about 8 mg of fine particles of Au-198 into the test loop and collecting them on a filter screen. These initial tests demonstrated that the system had adequate sensitivity. Figure 5 is a photograph of one of the test loops showing detectors, instrumentation, and the recorder.

Contaminant Tests

Several test materials were irradiated to determine their suitability for use with radioactive tags. They included:

    AC coarse test dust
    AC coarse test dust with iron pyrite
    AC fine test dust
    AC coarse test dust with zinc sulfide
    A cryogenic insulting material (POP)

Neutron activation analysis and gamma spectroscopy indicated that each of these materials contained trace elements suitable for use as a radioactive tag. All of the test materials showed radioactive Na-24 one day after irradiation. The tests indicated the presence of other isotopes, including Mn-56, Al-28, and Cu-64. No attempt was made to produce or detect long-lived radioisotopes for these tests.

Following these tests a sample of AC coarse dust was irradiated and then added to water flowing in a test loop. The loop contained a 325 x 2300 wire mesh screen as a filter. Data for a similar test using non-radioactive test dust were available from a previous test.

Typical results for one test are presented below:

    Test dust - AC coarse
    Add size   - adds weighing 4 milligrams each
    Run No.   - 2
    Screen   - 325 x 2300

Component	Dust mass (milligrams)
Filter screen	26.40
O-rings	0.48
Wiped from flanges	0.35
Presume deposited in test loop	0.77
	28.00

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Figure 5:
COMPONENT TEST LOOP

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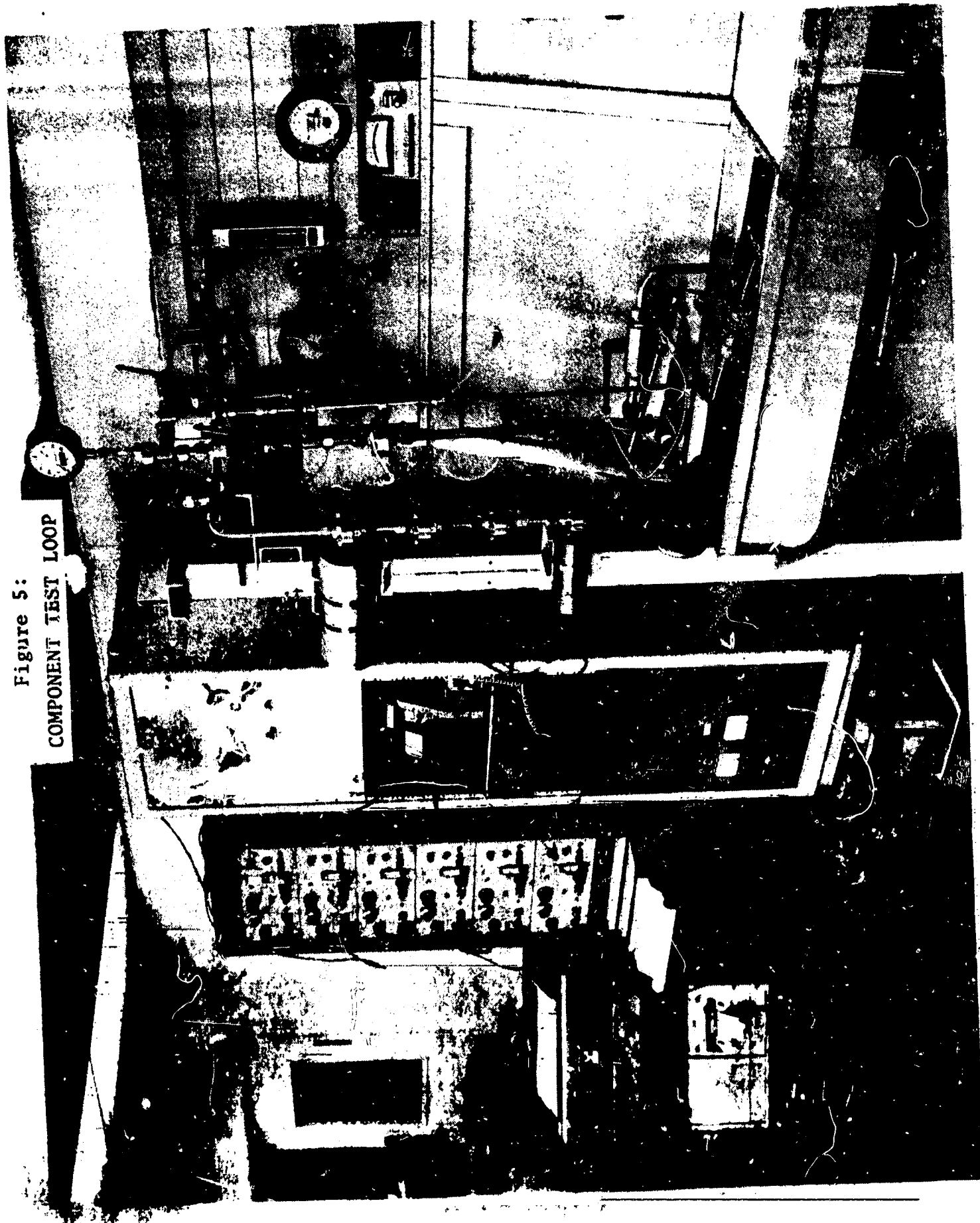
Analysis of the test results led to the following conclusions:

The radioactive method gave the same results as obtained previously using conventional methods. Comparison was based on the pressure drop across the screen vs. the mass of contaminant added. In the previous case the mass was determined gravimetrically, while in the radioactive case it was determined using the radiation detection system.

A careful investigation of the system revealed that some of the radioactive test dust was deposited on the internal walls of pipes, on the flange, and on O-rings used to seal the system, and never reached the filter screen.

Component Material Tests

Tests were also conducted to determine the suitability of certain materials as auto-tracers, specifically those which might be used for valve seats or bearing cages in cryogenic applications.

The materials tested under this portion of the program are shown in Table 5. The tests indicated that isotopes such as Na-24, Cu-64, Mn-56, Si-32 and others were present in every material. Figure 6 shows a typical gamma spectrum for one of the samples. Based on the test results, it was concluded that the autotracer method was also suitable for use with these materials.

TABLE 5: COMPONENT MATERIALS

Virgin Teflon
Kynar
Vespel
Teflon FEP
Nylatron GS
Kel F 81
Reprocessed Teflon
Nylon LP 410 6/6
Delrin
Virgin Teflon, 25% glass-filled
Fluorogreen E-600
Armalon
Rulon-J

Component Tests

Several components were tested during the course of the project. Results are summarized in Table 6. Figures 7 through 10 show the components tested. Some highlights from each test are described in the following paragraphs.

Solenoid Valves

Licensing limitations prohibited work with high level induced activity, thus causing the irradiation time to be relatively short. As the primary isotope developed was Mn-56 with only 2.58 hours half-life, the duration of the experiments was limited. Still, it was possible to detect less than 50 micrograms of material from the second valve, demon-

figure 6
GAMMA SPECTRUM OF
SAMPLE #3 - VESPEL

Cu-64
0.51 MeV

Mn-56
0.84 MeV

1.37 MeV

Na-24

2.75 MeV

Counts

Energy - MeV
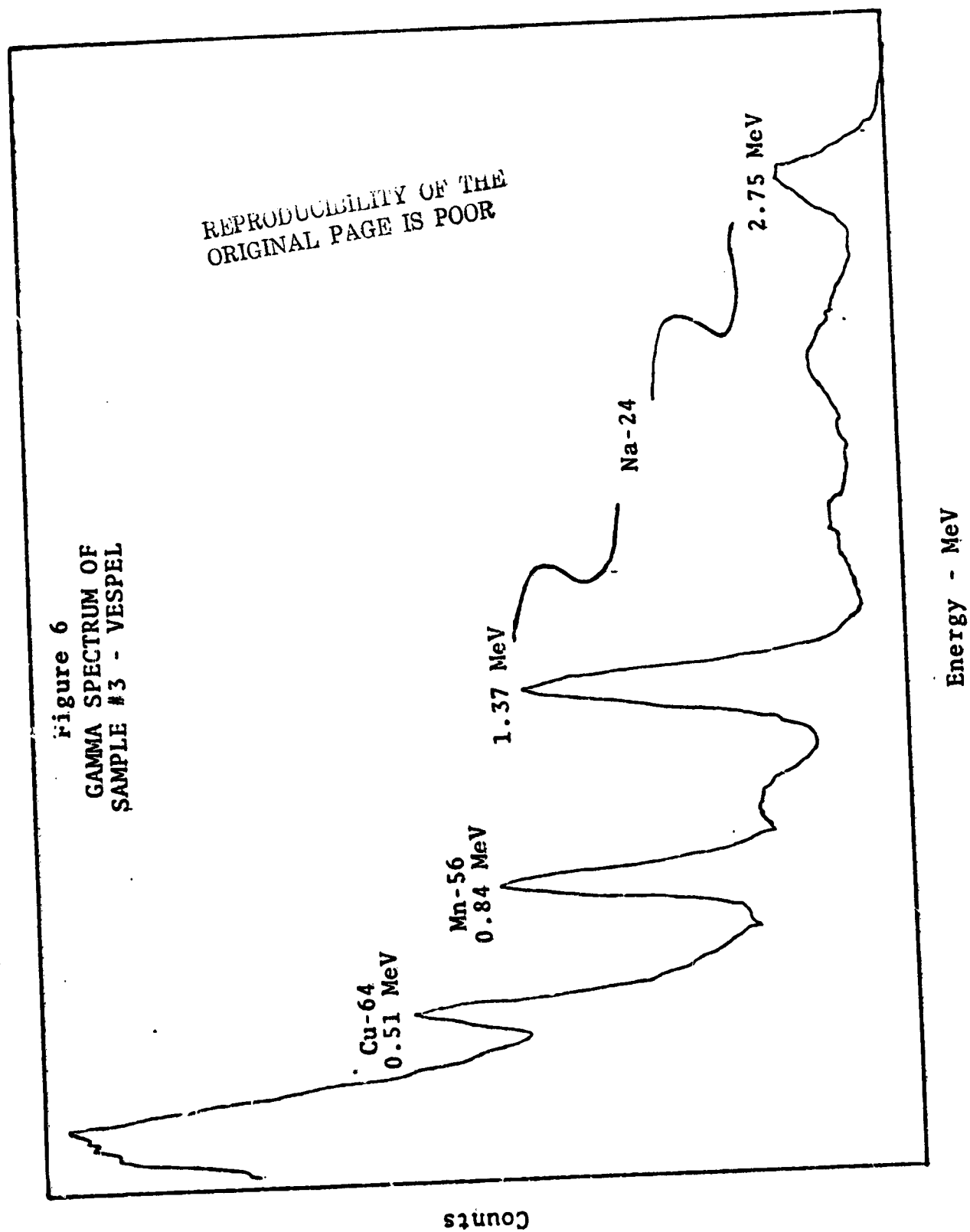
TABLE 6

COMPONENT CONTAMINANT GENERATION TESTS

COMPONENT	TEST FLUID	NUMBER OF CYCLES	CONTAMINANT GENERATION DETECTION
Solenoid Latching Valve	Water	5,000	Not detected
Solenoid Valve	Water	10,000[a]	<50 micrograms
Valve Assembly, augmented spark igniter	LN_2	1,700	4.8 milligrams
Hydraulic Actuator	Hydraulic fluid Mil-H-5606	5,400[b]	0.69 milligrams
Cryogenic bearings[c]	LN_2	240,000[d]	81.5 milligrams

NOTES:

(a) Test was run for 20,000 cycles total. Forty milligrams of AC coarse test dust was added during second 10,000 cycles; no radio-activity was detected due to decay.-

(b) Actuator was cycled 3,600 cycles unloaded and 1,800 cycles with a 5 lb side weight.

(c) Bearings were operated in a bearing test assembly.

(d) Test terminated due to failure of one set of bearings. .

CAUTION

RADIOACTIVE MATERIALS
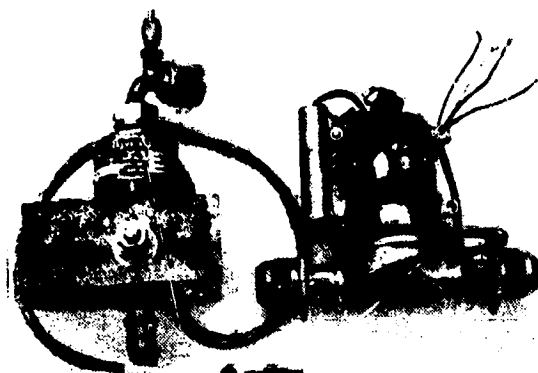
SOLENOID VALVES

FIGURE 7: SOLENOID VALVES

CAUTION

RADIOACTIVE MATERIALS
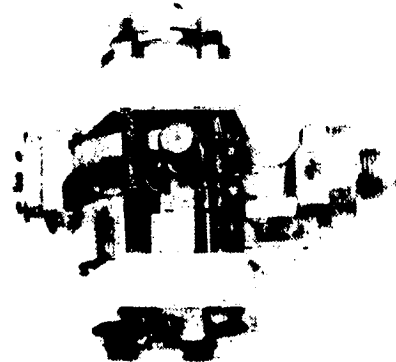
ASI VALVE ASSEMBLY

FIGURE 8: ASI VALVE ASSEMBLY

FIGURE 9: HYDRAULIC ACTUATOR
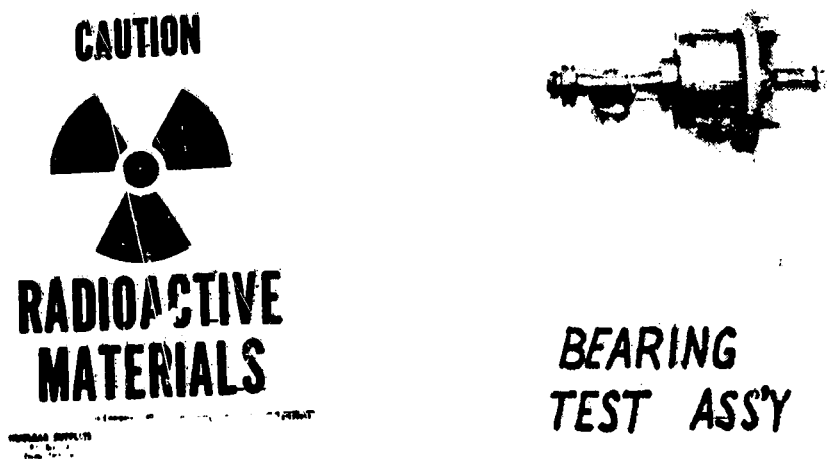
FIGURE 10: BEARING TEST ASSEMBLY

strating the sensitivity of the method.

Valve Assembly, ASI

This valve was unique in that it had a large aluminum body. To circumvent the high induced activity which otherwise would have been associated with irradiation of the body, it was shielded to prevent neutron activation. A cooling period was used before beginning the tests to allow the short-lived Al-28 to decay.

Analysis of the results indicated that 35% of the contaminants collected were on the 80 x 700 mesh screen, while 65% was collected on the 325 x 2300 screen. Since the dominant isotope was Cu-64, the contaminant is assumed to have originated from the valve body.
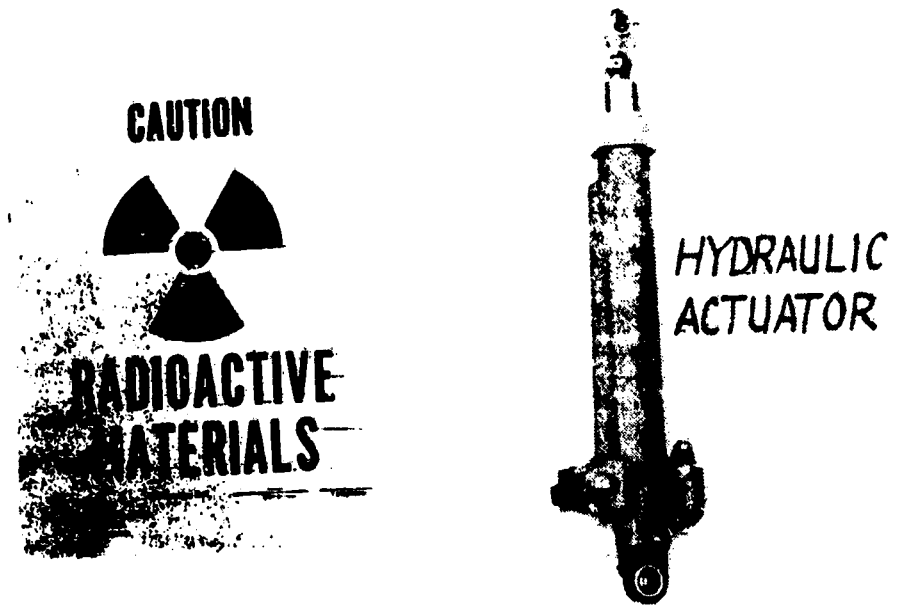
Hydraulic Actuator

This was one of the most interesting tests because it was feasible to allocate the distribution of contaminants between the body of the actuator and the bushing. The tests indicated that about two-thirds of the contaminants came from the actuator body, while the balance came from the bushing. Figure 11 shows the mass of collected contaminants as a function of the number of cycles of operation. The coarse screen (80 x 700 mesh) had only 2.5% of the total mass; 97.5% of the contaminants passed through the coarse screen and were collected on the fine screen (325 x 2300 mesh).

Bearings

The bearing test was also interesting in that it was the only test in which enough contaminants were generated to activate the on-line monitoring system.

Although it had been planned to operate the bearings at 20,000 rpm for 3 hours, after 12 minutes of operation a sharp rise in activity was detected by the on-line monitor. At the same time there was an increase in the high frequency sound produced by the bearing test device and the tests were terminated. Figure 12 shows the buildup of contaminants as a function of time.

Subsequent analysis indicated that approximately equal amounts of contaminants were deposited on the coarse and fine screens.

Two sets of bearings were used. One set had a Rulon-J cage, while the other had a cage fabricated from K-monel. The K-monel was the only material having copper; and copper was the major isotope (along with Mn-56) detected. These results indicate that it was

22

FIGURE 11: CONTAMINANT GENERATION HISTORY OF THE HYDRAULIC ACTUATOR
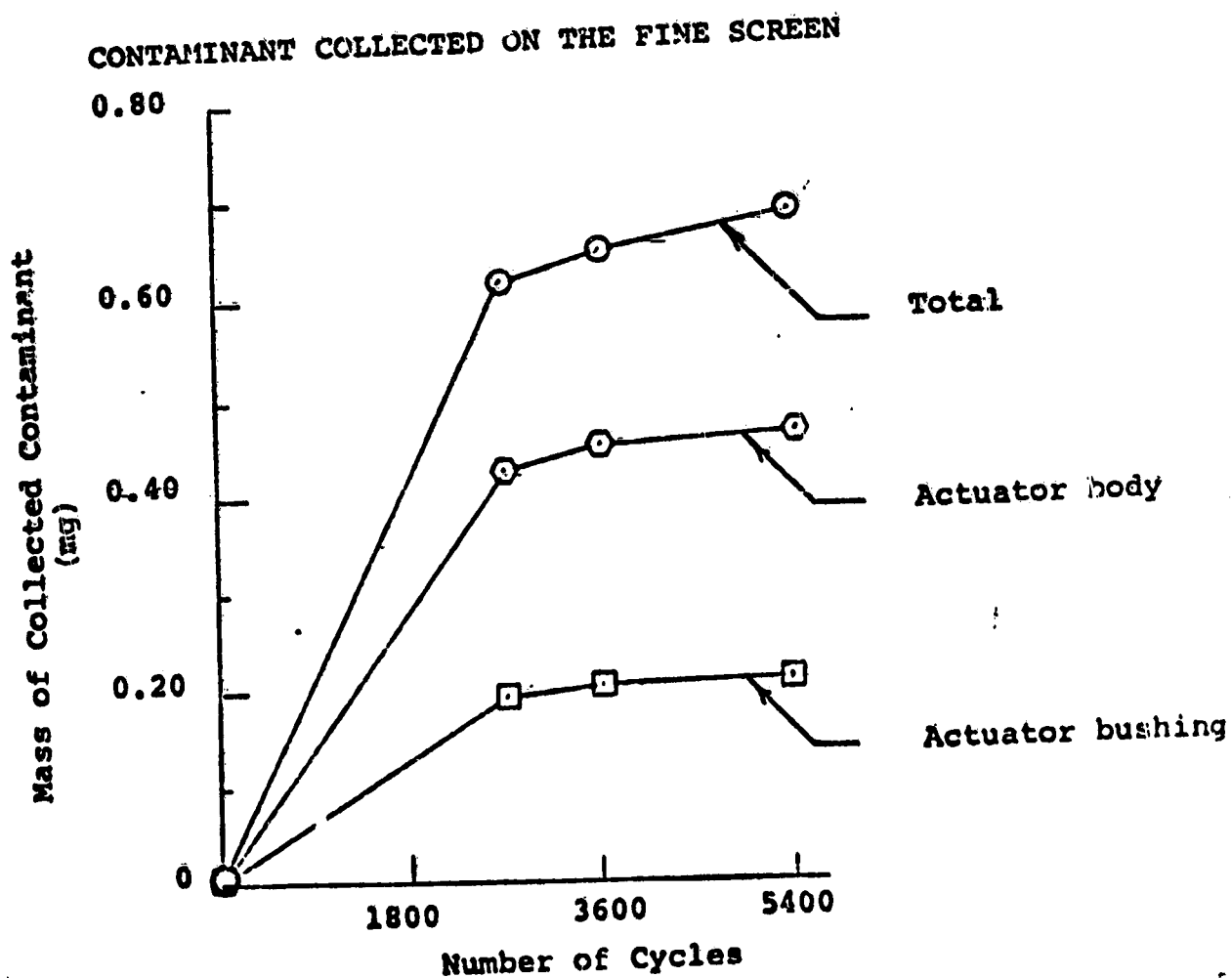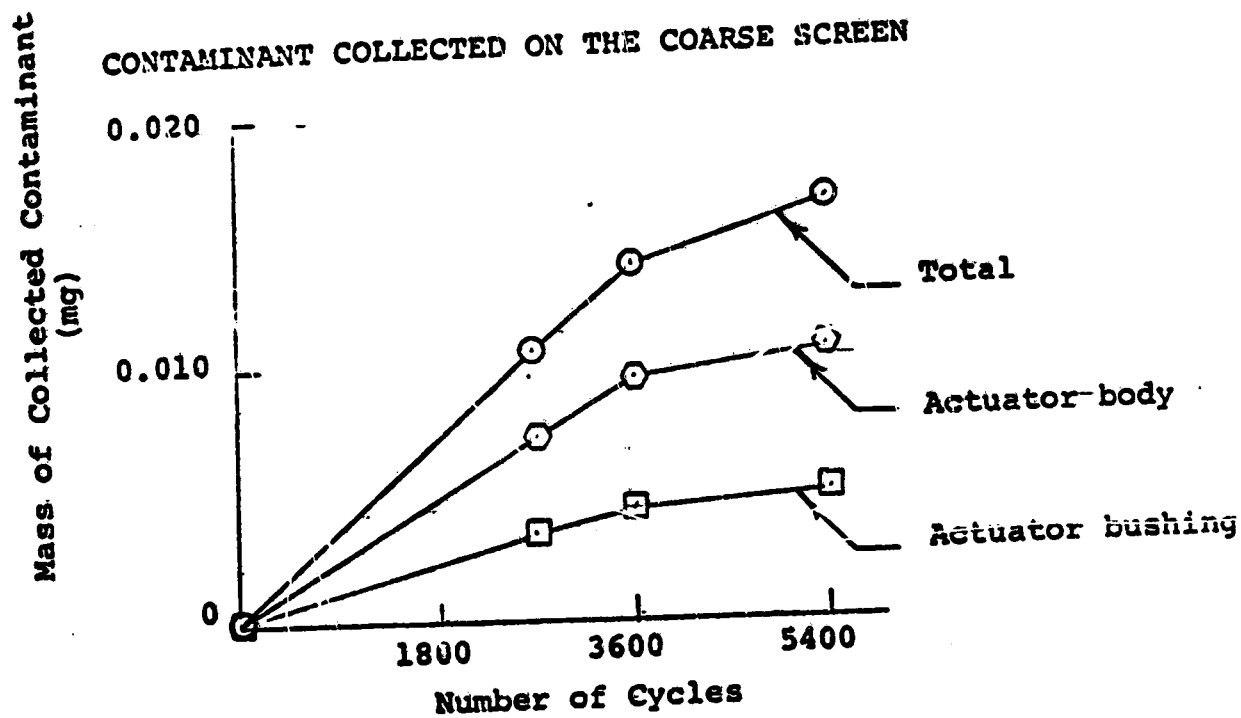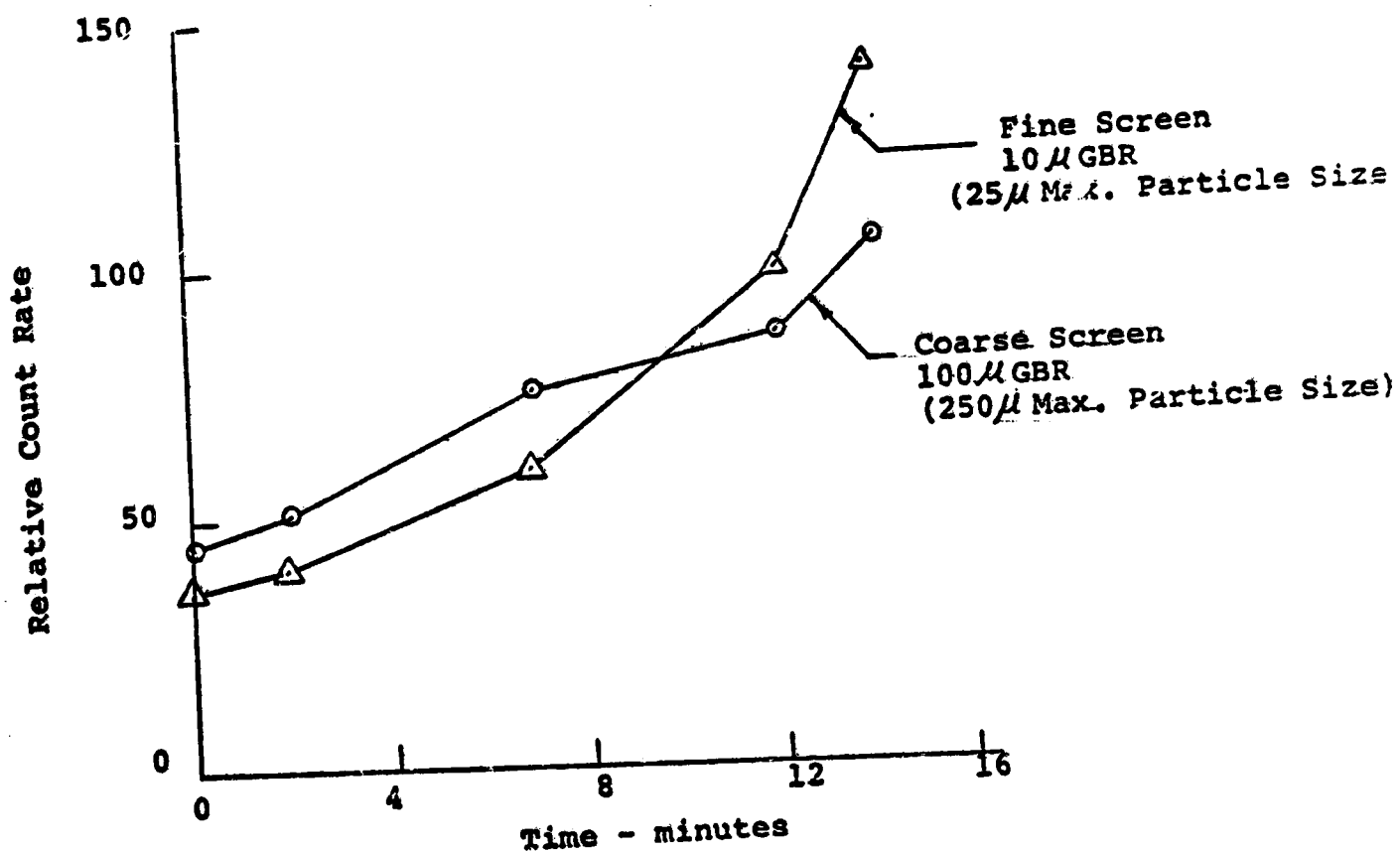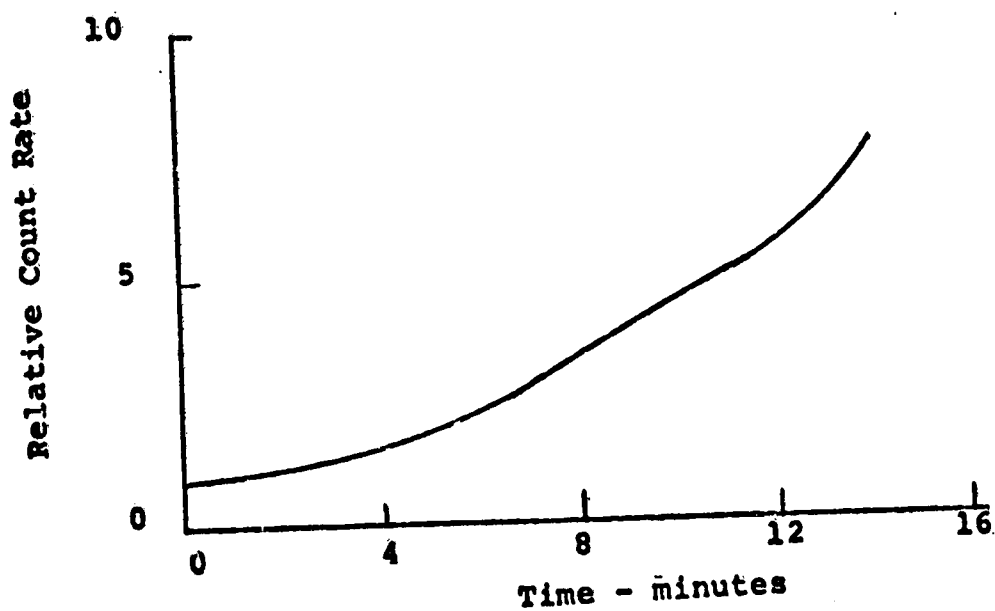
FIGURE 12:   CONTAMINANT GENERATION - BEARING TESTS

ON-LINE METER INDICATIONS

Fine Screen
10 μ GBR
(25 μ Max. Particle Size

Coarse Screen
100 μ GBR
(250 μ Max. Particle Size)

ON-LINE STRIP-CHART RECORD

the K-monel cage which yielded the major portion of the collected contaminant, although part of the contaminant was due to the Rulon-J cage and to the bearing races.

2.4 Conclusions

These tests have demonstrated the feasibility of using high sensitivity nuclear techniques to study contaminant generation in typical spacecraft components.

Tests have been conducted using test dusts, test materials, and typical components. The test results indicate that this method has several important advantages in addition to improved sensitivity: identification of contaminant sources, on=line measurements, and freedom from interference by non-test item contaminants. Results can be obtained in a relatively short time using conventional gamma spectroscopy instrumentation and radiation sources of moderate intensity.

3.0 GRAVIMETRIC ANALYSIS TECHNIQUE

3.0 Introduction

This section describes the technique for studying component contaminant self-generating characteristics in fluid systems where the expected amount of material to be released from the component exceeds 3 milligrams. The method has been in use for many years, but the inherent limitations of accuracy have not always been recognized. These limitations have been listed in Section 2.1 and are repeated below.

> Sensitivity is limited to 0.1 - 1.0 milligrams
>
> Discrimination between material generated by the test item and material released by the test system is difficult or impossible.
>
> The test system must be opened to determine the amount of contaminant collected.
>
> Certain fluids such as hydraulic fluids may create difficulty in obtaining accurate gravimetric results.
>
> Extreme care must be exercised to obtain accurate dry weight of the contaminant collection media before the test as well as obtaining accurate dry weight of the media and collected contaminant after the test.
>
> The multiple handling of the media and collected contaminant during drying and weighing can easily lead to loss of contaminant.

Where relatively large amounts of contaminants are expected, and extreme accuracy is not required, the gravimetric method of contaminant measurement is quite simple and requires very little special equipment and a minimum of personnel training.

As only one component, a 3/4" ball valve with Teflon seats and seal was expected to

generate a sufficient amount of material, the gravimetric technique was used on only this one test item.

3.2 Method

This method of evaluation of component contamination generation utilizes filter media installed downstream of the test component in a test system simulating actual operating conditions. As the test item is operated over a predetermined number of cycles, the test fluid flows through the component and carries generated material downstream where it is trapped or collected by a filter medium.

The collection filter medium is carefully weighed before and after the test. The weight difference is representative of the material collected on the medium during the test. Microscopic examination subsequent to weighing will provide information regarding the type and possible source of contaminant.

As the filter medium will collect all material larger than its filtration rating regardless of source, it is necessary that the test system and fluid be cleaned prior to testing a component. It is difficult to remove all system contaminant, so the test results are only approximations of the contaminant generated by the test item. For normally clean systems, the lower limit of contaminant generation for which the gravimetric technique will give the meaningful results is approximately 3 milligrams.

A typical test procedure for this technique is shown in the Appendix.

3.3 Results

A 3/4" ball valve was tested for contaminant generation in two test systems, one using water for the test fluid, the other using gas (nitrogen). As anticipated, a large quantity of contaminant consisting entirely of Teflon shreds was generated by the valve. In the test using nitrogen gas, 30 milligrams of material was collected, while the water test produced 60 milligrams of generated contaminant. The valve was cycled 1700 and 2700 times in the gas and water tests respectively. In both cases the Teflon contaminant consisted of shreds of material varying in size from 100 to 2000 microns long. Disassembly and visual examination of the internal parts indicated all of the material came from the stem seal which had extruded into the clearance space between the stem and body. Rotation of the stem in cycling sheared the extruded lip of Teflon allowing it to drop into the cavity between the ball and body. Further valve cycling then "swept" the loose material into the flow stream. There was no visible sign of wear or damage to the main seals.

3.4 Conclusions

The gravimetric technique can be used effectively when large amounts of contaminant are collected. Undoubtedly some material from the flow system was also collected on the 10 micron screen, but the percentage error caused is reduced due to the large amount of material generated by the component.

4.0 CONCLUSIONS AND RECOMMENDATIONS

The nuclear irradiation - autotracer measurement techniques for determination of contaminant generation characteristics have been proven to be practical and accurate. In addition to the obvious improved sensitivity of measurement there are several additional advantages over the gravimetric method; identification of contaminant sources, on-line measurement capability, insensitivity to contaminants other than those originating in the test item. Results can be obtained in a relatively short time using conventional gamma spectroscopy instrumentation and radiation sources of moderate intensity.

It is recommended that for determination of relatively small amounts of generated contaminant the autotracer technique be employed. For relatively large amounts of contaminant and where the system fluid can be filtered to a high cleanliness level, the gravimetric-microscopic analysis method can be used.

5.0 APPENDIX

TEST PROCEDURE FOR

CONTAMINANT GENERATION MONITORING USING AUTO-RADIOTRACER TECHNIQUES

1.0 Scope

This procedure describes the test method and equipment to be used in the application of auto-radiotracer techniques in monitoring contaminant generation in operating system components. The general procedures outlined may be applied, with appropriate modifications to any component in which contaminant generation is expected to occur.

2.0 Procedures

2.1 Material Identification and Activity Requirement

The first step in planning a contaminant generation test is to determine which parts of the given component are expected to wear, the constituent elements present in the critical parts, and the isotopes and corresponding half-lives which will result from irradiaiton. If the test is to be feasible at least one of the constituent elements must yield an isotope with sufficiently long half-life to allow completion of the planned test and still yield measurable quantities of radioactive material. Based upon this information, the irradiation conditions (neutron flux and duration) required to provide the desired monitoring sensitivity may be determined. The component parts should also be irradiated under identical conditions, to be used later as calibration standards in analyzing the test results.

2.2 Test Operation

The irradiated parts are reassembled and the component is installed in a test loop. Downstream of the component a series of progressively finer mesh filter screens should be placed. Test conditions should simulate actual operating conditions: system fluid, pressure, flow rate, temperature, cycling rate, and total cycles. At each filter position, a nuclear radiation detector (GM tube, scintillation detector) should be located; each detector having an individual meter readout and continuous strip chart channel to record the count rate as a function of cycles or wear time. Replacement screens should be prepared prior to the test to be used if needed. The component is then operated according to its operating specifications and the planned test procedures.

2.3 Measurement of Contaminant Generation

On-line monitors can be used to continually measure the buildup of generated contaminant

on the filter screens. Using the irradiated calibration standards, the various detector channels may be calibrated to provide a direct indication of the mass of generated contaminant being collected. Following the test, the filters are removed from the test loop and are counted in a sensitive multichannel analyzer gamma spectroscopy system.

3.0 Analysis of Results

The on-line monitors provide an indication of the amount of generated contaminant being collected on the screens. The relative activity levels on the various screens give an indication of the particle size distribution of the generated contaminants.

The results of the multichannel analysis provide a precise determination of the mass and nature of the generated contaminant. Comparing the screens to the calibration dards, one may determine the amount of contaminant being generated from each of seve parts of the component being tested. Quantities of material of 1 gm can be detected this manner. This information may then be used to analyze the behavior of the component during operation and identify those parts which generate contaminants.

TEST PROCEDURE FOR

CONTAMINANT GENERATION MEASUREMENT BY THE GRAVIMETRIC METHOD

1.0 Scope

This procedure describes the test methods and equipment to be used in the application of gravimetric techniques to the determination of contaminant generation characteristics of components.

2.0 Procedure

2.1 System and Component Preparation

Prior to testing the component and the flow test system must be cleaned to the cleanest practical level. The cleaner the system and test fluid, the more meaningful will be the test results. The component should be cleaned to at least the level specified for flight hardware. The fluid and test system may be cleaned by circulating the test fluid with a fine filter installed immediately upstream of the test item installation point. The "clean up" filter should have a glass bead rating of 10 microns or finer.

2.2 Collection Filter Preparation

The filter medium that will be used for collection of generated contaminant must be clean and dry prior to weighing. The filters should be sized to collect all particles of interest. Thus if particles below 10 microns in size are to be evaluated as well as larger particles, the membrane type of media should be used. For particles above 10 microns in size, one of the various standard filter screens may be used in accordance with the following table.

To Collect Particles Above Size Shown	Use Following Medium
10 Microns	325 x 2300 Twilled Dutch Double Weave
20 Microns	2 x 120 x 650 Plain Dutch Single Weave
40 Microns	80 x 400 Plain Dutch Single Weave
100 Microns	30 x 150 Plain Dutch Single Weave

After cleaning and drying the filter medium the filter cloth is weighed to the neare 0.1 milligram and installed in a suitable holder.

3.0 Test Operation

The test item is mounted in the fluid system with the pre-weighed collection filter mounted as close as practical to the component outlet. With the test fluid flowing

through the test system the component is actuated for a predetermined number of cycles in accordance with its normal operating specification and planned test procedure.

4.0 Analysis of Results

At the conclusion of the test, the collection filter medium is carefully removed from its holder and dried to a constant weight. The difference between the pre-test and post-test weight is representative of the amount of contaminant generated by the test component. Further analysis of the type and size distribution of the generated material may be conducted using normal microscopic techniques.

CONTAMINATION GENERATION STUDIES

SPECIFIC TEST DESCRIPTIONS

TEST NUMBER: 1

TEST ITEM: Parker Solenoid Operated Latching Valve

1.0 Scope

The purpose of this test was to determine the contamination generation characteristics of a solenoid operated isolation valve. The test item was a solenoid valve manufactured by Parker Hannifin and used on the RCS system of the Apollo CSM. The test was designed to determine the contamination generation rate of the Parker valve as a function of the number of cycles of valve operation in a flowing liquid test system. The test procedure involved irradiating the valve and then tracing radioactive material worn from the valve in a test flow loop by collecting particles on four filters arranged in series. For this test, no attempt was made to study specific parts of the valve (i.e., the valve seat).

2.0 Procedure

The entire valve was irradiated for 30 minutes using a neutron generator having a yield of 10^{11} neutrons per second at an energy level of 14 MeV. A gamma spectrum of the valve was then recorded and is shown in Figure 1-1.

The irradiated valve was installed in test loop containing a pump, flow control valve, and four grades of collection filter media mounted in series downstream of the test item. The collection filters consisted of 3 stainless steel wire cloth discs and a Millipore 5 - micron membrane filter. The micron rating of the collection filters was as follows:

LOCATION	TYPE	MICRON RATING (GBR)
1st Filter	80 x 700 TDDW	40 Microns
2nd Filter	165 x 1400 TDDW	20 Microns
3rd Filter	325 x 2300 TDDW	10 Microns
4th Filter	Millipore Membrane	5 Microns

A Geiger - Mueller tube was placed in a fixed position to monitor the Millipore filter, while a scintillation detector with adjustable position was used to monitor the 3 other filters.

A control system was provided to automatically cycle the solenoid-actuated test valve and to count and record the number of cycles through which the valve had been operated.

The test fluid was deionized water pre-filtered to 0.45 microns.

Figure 1-2 shows the test system schematic.

Background radiation levels were measured at the four filter positions before valve cycling was initiated. Then, in increments starting at 20 cycles and ranging up to 500 cycles, the valve was actuated. Following each valve cycling increment, radiation levels were measured at the four filter positions and recorded on a six-channel Sanborn recorder. The flow rate was held at 4.0 gpm throughout the experiment and the system pressure was recorded just before each valve cycling. The millipore filter was replaced twice during the experimental run: at 1800 cycles and at 3000 cycles. The valve was actuated through 5000 cycles with data taken at 37 points.

Data collected throughout the run showed no statistically significant variations in the count rate at any of the four filter positions. This indicated that no _radioactive_ material was being worn off the valve during cycling. This finding was confirmed following the test in which the valve was cycled 5000 times. When the filters were removed from the test loop and counted in a shielded counting pig, no activity levels significantly above background were detected.

It was noted that as the run progressed, the pressure drop in the system continually increased. Each time the millipore filter was replaced, the pressure drop decreased to its initial level of 107 psig, (Figure 1-3), but increased again as the run proceeded. The maximum pressure drop reached was 142 psig. This indicated that _something_ was being trapped on the millipore filter, either dirt from the system or _non-radioactive_ material being worn from the valve. The latter is a definite possibility since the valve seat is made of teflon which is not detectable by _this_ technique. Visual inspection of the millipore filters clearly showed that something was being trapped on them. Microscopic analysis of the material trapped on the filters was performed to determine the nature and the source of the material which was trapped on the filters. A description of particles found follows.

Microscopic Analysis of Filters

Analysis of material collected downstream of irradiated Parker propellant valve, tested June 29, 1971.

80 x 700 Screen (40μ) Filter Number 1

MATERIALS	SIZE
	50 x 740 micron
Plastic Fibers	

Particles

Orange Colored

Bright Orange, Plastic-like

Dull Grey Material

Clear Teflon Shreds   470 x 250 Micron

Copper Colored Material   190 x 125 Micron

Lemon-Orange Plastic   150 x 100 Micron

Concentration of Particles - Including:

Fibers, Teflon, Grey Slag, Clear Plastic,

Red Colored Spongy Material, Copper Colored Material

165 x 1400 Screen (20μ) Filter Number 2

MATERIALS	SIZE
Ruby Colored Transparent	
Milk Colored Plastic Globule (square with rounded corners)	250 x 190 Micron
	500 x 12 Micron
Fibers	125 x 60 Micron
Copper Colored Material	
	190 x 125 Micron
Tan Plastic	430 x 100 Micron
Metal Sliver (Burned colors are blue, red, amber, yellow)	
	250 x 190 Micron
Grey Metallic	125 x 100 Micron
Brick Colored - Sharp Corners (Several were noted within small region)	

325 x 2300 Screen (10μ) Filter Number 3

MATERIALS	SIZE
Copper Colored	
Black, Charcoal-like	260 Micron
Grey Colored	
Plastic Fiber	300 x 25 Micron

Millipore (5 Micron) - Filter Number 4

Sample Number 1: 1200 Cycles, Millipore Broken

    Many large shavings of grey metallic-like appearance, also

    some smooth metallic particles - copper colored gold or brass colored

Sample Number 2: 3000 Cycles

    Metallic (250 Micron)

    Charcoal-like cluster (600 x 500 Micron)

Predominance of silver colored shavings, (Metallic)

Plastic (125 x 60 Micron)

Sample Number 3: 5000 Cycles

Small amounts of grey metallic particles

Shiny metallic particles

Plastic

Bronze colored material

Conclusions

As the predominance of material found on the filters consisted of plastic type particles there is an indication that the Teflon seat of the valve is the probable source. The irradiation level of the valve was such that the seat material would not have become active enough for detection by the gamma ray monitors. The absence of indications of radioactivity on any of the four filters shows that the metallic particles observed in post-test microscopic analysis originated in the test system and were probably generated by assembly of threaded connections.

The combination of microscopic analysis and nuclear detection techniques shows the ability to detect the generation of non-radioactive material (by visual observation) and to discriminate between metallics originating in the test system itself (non-radioactive) or the test item (radioactive). The organic particles cannot definitely be proven to have originated in the valve seat, but their presence in the collection screen gives cause for further investigation of the seat wear characteristics.

Although the system was cleaned prior to the test, the violent shaking of the system caused by water - hammer with valve shut off at 4 gpm flow will dislodge particles trapped in crevices within the test loop. The advantages of being able to discriminate between these particles and those originating in the test article are obvious.
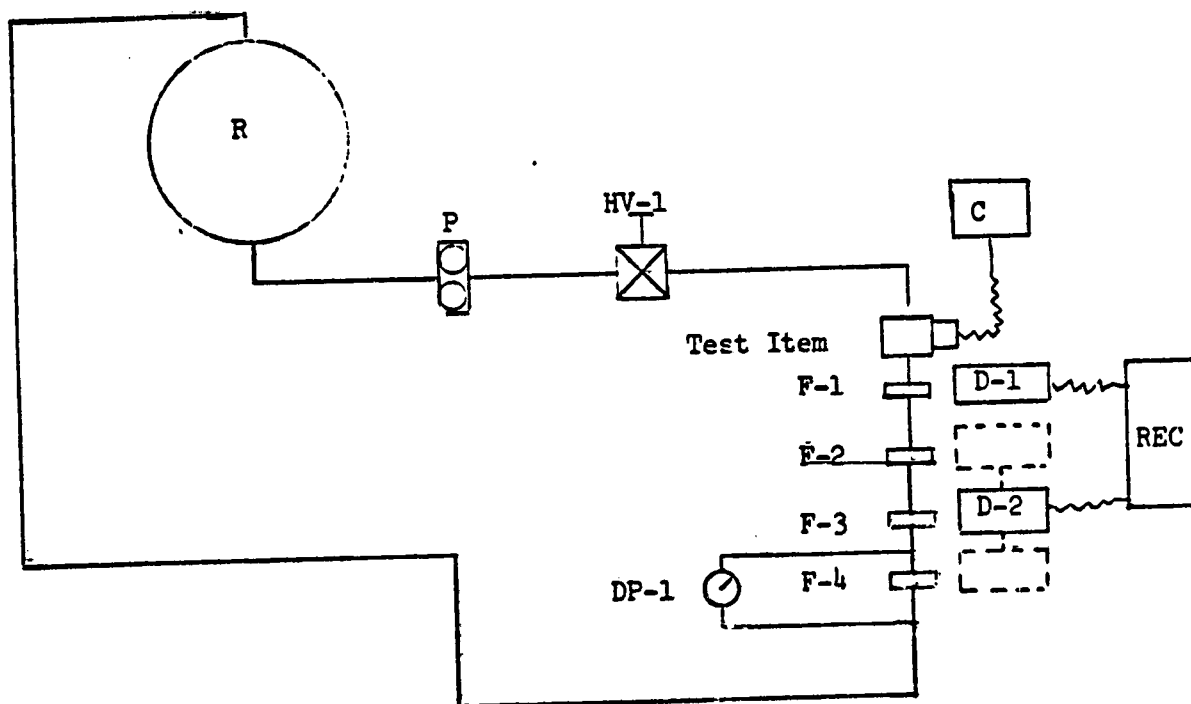
FIGURE 1-1

GAMMA SPECTRUM - IRRADIATED PARKER VALVE
30 MINUTES FAST NEUTRON IRRADIATION
ON JUNE 8, t_0 = 1050

REPRODUCIBILITY OF THE
ORIGINAL PAGE IS POOR
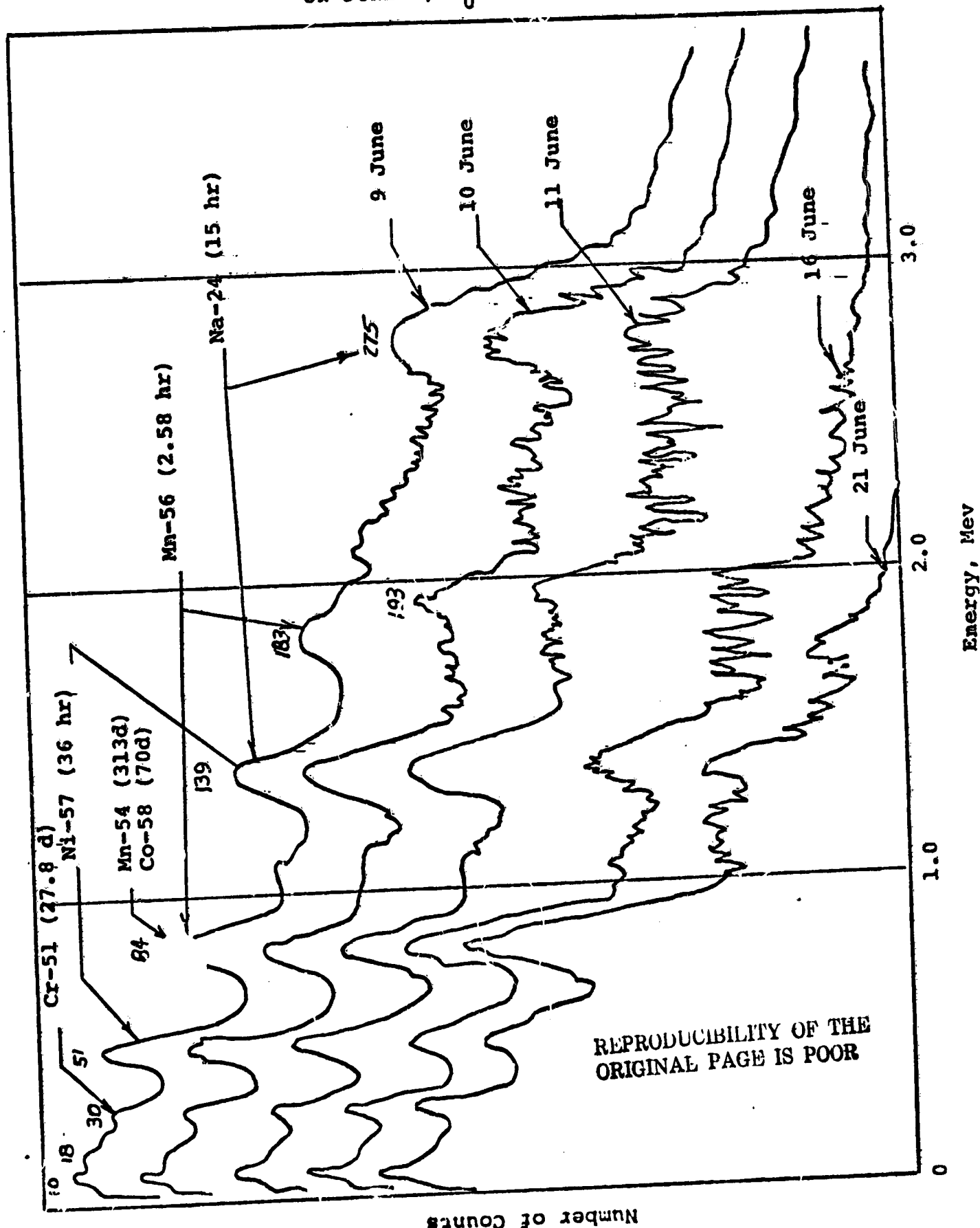
Number of Counts

FIGURE 1-2

Contamination Generation Test System Schematic

R - Reservoir
P - Pump
HV -1 - Flow Control Valve
F-1 - Collection Filter 80 X 400 Twilled Dutch Double Weave (40μ)
F-2 - Collection Filter 165 X 1400 Twilled Dutch Double Weave (20μ)
F-3 - Collection Filter 325 X 2300 Twilled Dutch Double Weave (10μ)
F-4 - Collection Filter Millipore Membrane   (5μ)
D-1 - Geiger Mueller Tube Detector
D-2 - Scintillation Detector Movable to 3 positions
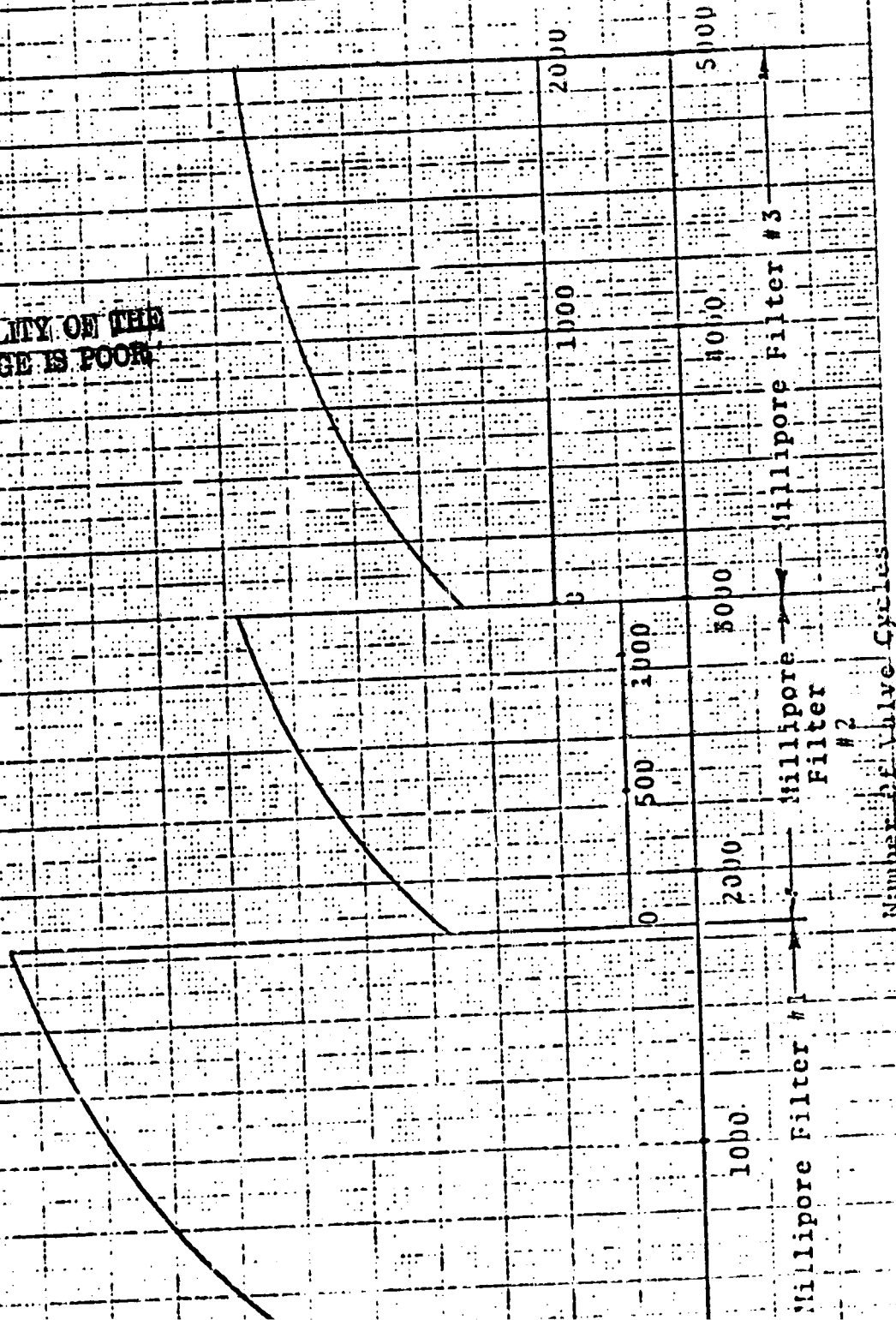DP-1 - Differential Pressure Gage
C - Controller
Rec - Six Channel Sanborn Recorder

FIGURE 1-3
SYSTEM PRESSURE DROP DURING WEAR TEST
OF THE PARKER LATCHING SOLENOID VALVE

TEST NUMBER: 2

TEST ITEM: Rocketdyne J-2 Augmented Spark Igniter (ASI) Valve

1.0 Scope

The purpose of this test was to determine the contaminant generating characteristics of a Rocketdyne pneumatically operated Augmented Spark Igniter (ASI) Valve as used on the J-2 rocket engine. The valve is designed for use in a cryogenic system (LOX) and the test was conducted by cycling the valve in a flowing system of liquid nitrogen. A cross-section drawing of the valve is shown in Figure 2-1.

2.0 Procedure

The valve was tested under cryogenic conditions using liquid nitrogen as the test media. The valve was leak tested prior to the flow actuation test, cycled through 1700 cycles with 100 psig inlet pressure LN_2 and leak tested again at the conclusion of the test. During the "valve open" portion of each cycle the liquid nitrogen flow rate was approximately 9 gpm. Particulate matter generated by the valve was collected on two downstream filters of 40 and 10 microns glass bead rating and measured using autotracer techniques.

The procedures followed in testing the Rocketdyne valve included the following steps:

1. Disassembly of the valve and fabrication of parts to facilitate quick reassembly.
2. Irradiation of the critical-wear components of the valve.
3. Reassembly of the valve.
4. Leak testing and valve cycling with LN_2 flow followed by leak testing.
5. Analysis of the test results.

Care had to be taken in disassembling the valve to prevent damage to the valve bellows. The critical parts of the valve were identified as the valve body and the seat and poppet assembly. In order to remove these parts, it was necessary to cut several welds. To eliminate re-welding the valve before testing, assembly brackets were designed and fabricated. These brackets provided leaktight sealing of the valve without the need for welding upon reassembly.

the valve critical to wear, the seat and stem were surrounded by cadmium. This left
exposed only the seat and stem and that portion of the valve body immediately surround-
ing these parts. The cadmium shield effectively reduced the mass of aluminum exposed
to the neutron flux upon irradiation from approximately 1000 to 100-gm. Based upon
the activation calculations and the flux depression considerations (presented in the
following "Pre-Irradiation Activity Calculations") it was concluded that activation of
the components in the thermal column of the UCLA reactor for 1 hour at 100 Kw, and at
a point where the normal undepressed flux is 5×10^{10} n/cm^2 sec, would yield a total
activity (after a 1 hour decay period following reactor shutdown) approximately equal
to 9.5 mCi, the activity limit permissible by the license under which the test was
performed. The actual activity attained was measured to be approximately 7 mCi upon
removal from the reactor, 1 hour after the reactor had been shut down.

Following activation of the body, seat, and stem the valve was reassembled and instal-
led in the test loop. As indicated previously, the reassembly was facilitated by the
special brackets which had been fabricated earlier. This simplified reassembly method
(rather than re-welding the valve) was beneficial in two ways: first, the reduced assem-
bly time resulted in less decay time and subsequently a higher initial activity level
at the start of the test; second, due to the reduced time required to reassemble the
valve, personnel exposures during this operation were also reduced.

The assembled valve was installed in the test loop which consisted of a bank of dewars
containing LN$_2$ upstream of the valve. Downstream of the valve were installed an 80 x
700 mesh screen, a 325 x 2300 mesh screen, and a vent. The valve actuation mechanism
was a remotely controlled pneumatic system. A solid-state scintillation detector placed
in a collimating shield was located at each filter position. Additional lead shielding
was placed around the activated valve to further reduce the background level at the
filter positions. Figure 2-2 shows the test system in the operating mode.

The valve was then operated through 1700 cycles with 100 psig inlet LN$_2$ pressure and 9
gpm flow while open. Following the completion of the test, the two screens were removed
from the test loop and counted using a sensitive sodium iodide scintillation crystal and

is shown in Figure 2-5. The count rates for the two screens and background were as follows:

80 x 700 screen	514 counts/40 min.
325 x 2300 screen	676 counts/40 min.
Background	326 counts/40 min.

The net count rates are therefore,

80 x 700 screen	188 counts/40 min. = 4.70 cpm
325 x 2300 screen	350 counts/40 min. = 8.75 cpm

These count rates were determined from the integrated counts under the Cu-64 gamma peak which was the predominant peak in the gamma spectra. The two screens and the standard were counted with in a period of 2-3 hours. Compared to the 12.8 hr. half-life of Cu-64, little relative decay occurred between the samples counted over the time required to complete the three counts. The results may therefore be directly compared without correcting for radioactive decay.

The activity standard consisted of 1.5 mg of material taken from the activated valve body. In a 40 minute count, this sample yielded 493 counts, or 167 counts/40 minutes = 4.17 cpm above background. The specific activity of the standard is therefore:

$$\frac{4.17 \text{ cpm}}{1.5 \text{ mg}} = 2.8 \frac{\text{cpm}}{\text{mg}}$$

Using this specific activity and the count rate data presented, the amounts of generated contaminant collected on the screens are given by:

80 x 700 screen: $\frac{4.70 \text{ cpm}}{2.8 \text{ cpm/mg}} = 1.7 \text{ mg}$

325 x 2300 screen: $\frac{8.75 \text{ cpm}}{2.8 \text{ cpm/mg}} = 3.1 \text{ mg}$

The two screens were weighed before and after the test, yielding the following amounts of material collected on the filters during the test:

80 x 700 screen:	2.4 mg
325 x 2300 screen:	1.2 mg

In the case of the 325 x 2300 screen, there is an apparent discrepancy between the weight of material calculated by gamma ray radiation and simple gravimetric analysis. It would be not surprising to find a larger weight by gravimetric analysis as all material collected from the system itself would be included. The lesser weight by gravimetric determination is attributed to handling loss between the time of gamma analysis and final

10 microns in diameter, dark silica-like material, metallic particles up to 60 microns in size, and several white plastic appearing particles up to 100 microns.

The preliminary gamma spectra disclosed the presence of copper - 64 created from the copper constituent (.15 to .40 percent) in the 6061 aluminum body. This material was apparently "scuffed" from the body bore by the sliding chrome-plated poppet.

Internal leakage tests conducted on the A.S.I. Valve using nitrogen gas at 100 psig showed the assembly to be "bubble tight" both before and after the test.

3.0 Conclusions

This test demonstrates the feasibility of using radioactively tagged components to measure contamination generation of critical components in more complex systems.

PRE-IRRADIATION ACTIVITY CALCULATIONS

Rocketdyne ASI Valve

The details of the calculations to determine the potential activity and required irradiation time for the valve seat and stem are shown in Table 2-1, and the calculations for the valve body are shown in Table 2-2.

Total activity obtained irradiating both components at the same point in the thermal column is given as:

47.1 mCi	Valve seat and stem
6.1 mCi	Valve body
53.2 mCi	Total (after 1 hr decay)

This value of 53.2 mCi is based on the flux in the thermal column with no flux depression. The valve body will be wrapped in Cd which will depress the flux. Assuming a flux depression factor of 5, the activity after 1 hour will be 10 mCi (approximately the radioactive materials license allowable limit). This is only an estimate. The irradiated parts may be left in the reactor for more than one hour before transferring the irradiated material if the parts come out of the reactor "hotter" than expected.

Immediately after 1 hour irradiation, the total activity is calculated to be:

94 mCi	body
650 mCi	seat and stem
750 mCi	

The dose rate is also calculated to be:

$$\frac{750 \text{ mCi}}{10 \text{ mCi}} = \frac{D \text{ mr/hr}}{60 \text{ mr/hr}} \quad \text{or} \quad D = 4.5 \text{ R/hr}.$$

i.e., the dose rate immediately after irradiaiton for 1 hour will be 4.5 R/hr. It is therefore suggested that the valve be left in the reactor to decay for 45 minutes after irradiation. By that time, the short half-live isotopes will have decayed and it will be possible to estimate how much additional decay time is necessary before the material may be transferred.

TABLE 2-1

ROCKETDYNE ASI VALVE P/N 308880, VALVE BODY PRE-IRRADIATION CALCULATIONS

(Valve body - 100 gm 6061 Al   t = 1 hr ϕ = 5 x 10^{14})

		m (gm)	MW (gm/gmole)	σ (x10^{24}cm^2)	$t_{1/2}$ (hr)	$1-e^{-\lambda t}$	A (μCi)	γ-activity (%)	$e^{-\lambda t}$	A (μCi)
Si-31	100(0.08)(0.03):	0.24	30	0.10	2.62	0.232	1.46x10^2	0	0.768	1.12x10^{2*}
Fe-59	100(0.07)(0.0033):	0.023	58	1.2	1080	~0	0	--	1	0
Cu-64	100(0.004)(0.70):	0.28	63	4.76	12.8	0.053	9.45x10^2	0.5	0.947	1.95x10^2
Mn-56	100(0.0015)(1.00):	0.15	55	13.3	2.58	0.235	6.80x10^3	100	0.765	5.20x10^3
Mg-27	100(0.012)(0.11):	0.13	26	0.3	0.16	0.987	1.15x10^3	100	0.013	1.50x10^{21}
Cr-51	100(0.0035)(0.0043): 0.0015		50	16	648	~0	0	--	1	0
Zn-69	100(0.0025)(0.186):	0.046	68	1	0.87	0.55	2.98x10^2	0	0.45	1.34x10^{2*}
Ti-51	100(0.0015)(0.053):	0.008	50	5.3	0.083	1.00	6.73x10^2	100	~0	0
Cu-66	100(0.004)(0.30):	0.12	65	1.9	0.085	1.00	2.79x10^3	9	~0	0
Al-28	100(0.96)(1.00):	96.0	27	0.23	0.038	1.00	6.40x10^5	100	~0	0
					TOTALS:		6.50x10^5†			6.11x10^3¶

NOTES: * ~ all β

¶ all β

† Activity at removal from the reactor

¶ γ-activity after 1 hour irradiation and 1 hour decay

The following is the equation used to calculate the activities:

$$A(\mu Ci) = \frac{mN_a\sigma\phi}{MW}(1-e^{-\lambda t}) \cdot \frac{1}{3.7 \times 10^4}$$
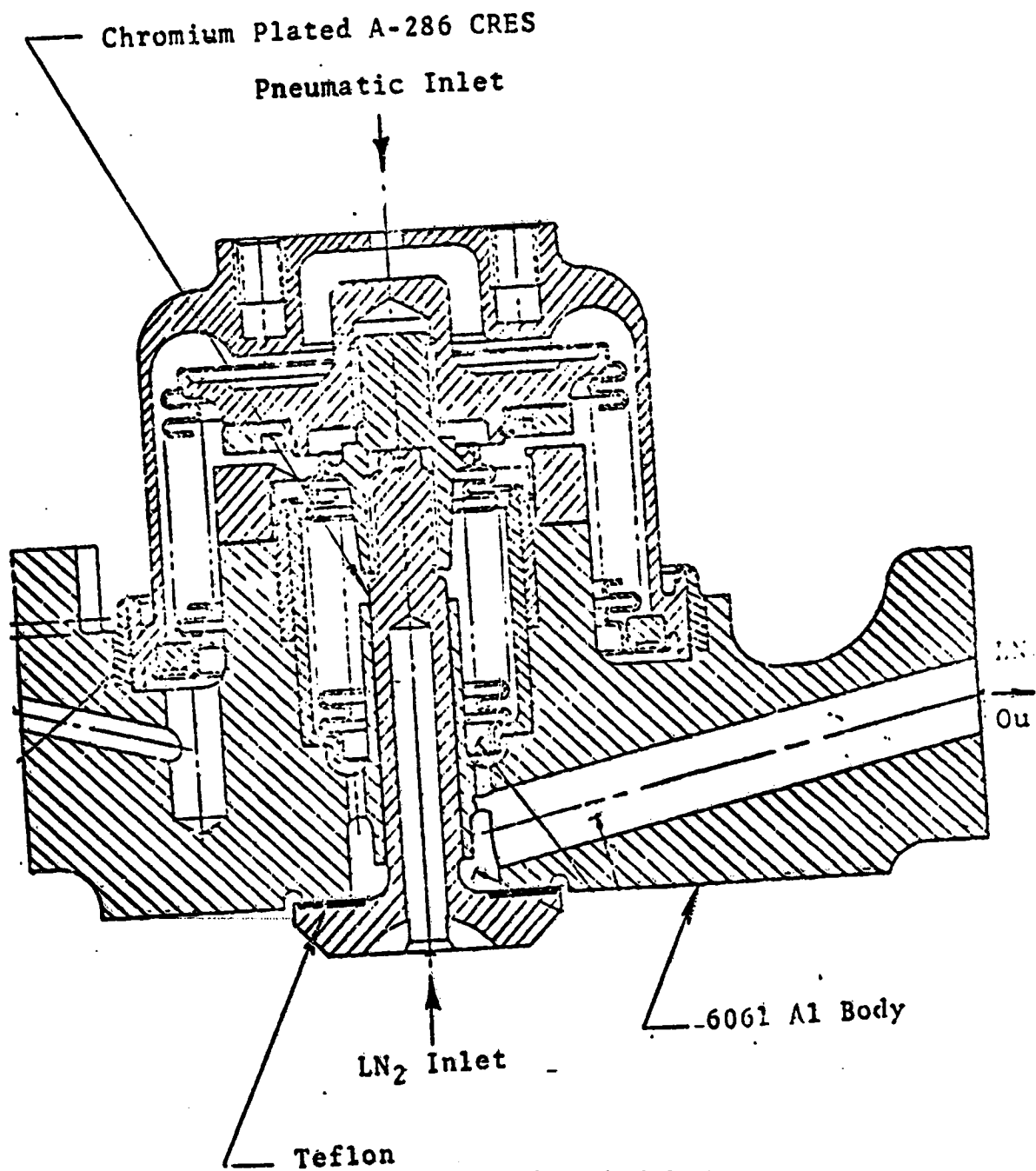
TABLE 2-2

ROCKETDYNE ASI VALVE P/N 308880, VALVE SEAT AND STEM PRE-IRRADIATION CALCULATIONS

(Valve seat and stem - 100 gm A-286   t = 1 hr ϕ = 5 x 10^{14})

		m (gm)	MW (gm/gmole)	σ (x10^{24}cm^2)	$t_{1/2}$ (hr)	$1-e^{-\lambda t}$	A (μCi)	γ activity (%)	$e^{-\lambda t}$	A (μCi)
Mn-56	100(1.00)(0.0135):	1.35	55	13.3	2.58	0.235	6.10x10^4	100	0.765	4.66x10^4
Si-31	100(0.03)(0.0095):	0.028	30	0.10	2.62	0.232	1.70x10^1	0	0.768	1.31x10^{1*}
Cr-51	100(0.0043)(0.15):	0.065	50	16	648	~0	0	--	1	0
Ni-65	100(0.0116)(0.26):	0.30	64	1.7	2.55	0.238	1.54x10^3	43	0.762	1.17x10^{3¶}
Ti-51	100(0.053)(0.0215):	0.11	50	5.3	0.083	1.00	9.24x10^2	100	0	0
V-52	100(1.00)(0.003):	0.30	51	4.9	0.062	1.00	2.33x10^4	100	0	0
Al-28	100(1.00)(0.002):	0.20	27	0.23	0.038	1.00	1.33x10^3	100	0	0
Mo-99	100(0.24)(0.0125):	0.30	98	0.14	66.6	0.01	0	--	0.99	0
Mo-101	100(0.10)(0.0125):	0.12	100	0.20	0.24	0.95	1.85x10^1	100	0.05	9.25
Fe-59	100(0.0033)(0.55):	0.18	58	1.2	1080	~0	0	--	1	0
					TOTALS:		9.37x10^4†			4.71x10^4¶

NOTES: * ~ all β

¶ 57% β

† Activity at removal from the reactor

¶ γ-activity after 1 hour irradiation and 1 hour decay

The following is the equation used to calculate the activities:

$$A(\mu Ci) = \frac{mN_a\sigma\phi}{MW}(1-e^{-\lambda t}) \cdot \frac{1}{3.7 \times 10^4}$$

Chromium Plated A-286 CRES

Pneumatic Inlet

6061 Al Body

LN$_2$ Inlet

Teflon

FIGURE 2-1

ROCKETDYNE J-2 ASI VALVE ASSEMBLY (P/N 308880)

FIGURE 2-2
ASI VALVE TEST

47

FIGURE 2-3

COARSE SCREEN GAMMA SPECTRUM

Cu-64

Log Counts

Energy Mev

FIGURE 2-4

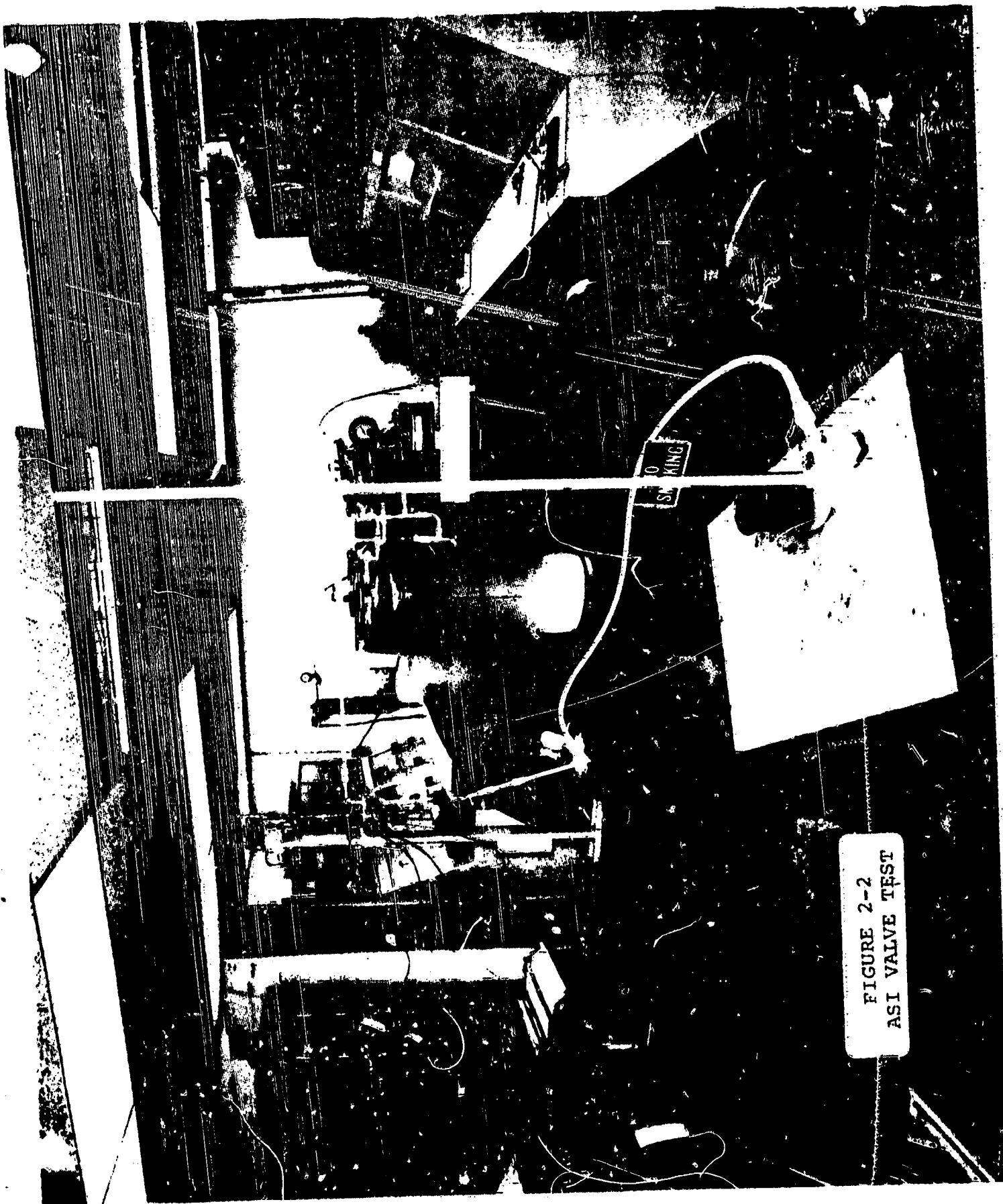FINE SCREEN GAMMA SPECTRUM

Cu-64

Log Counts

Energy Mev
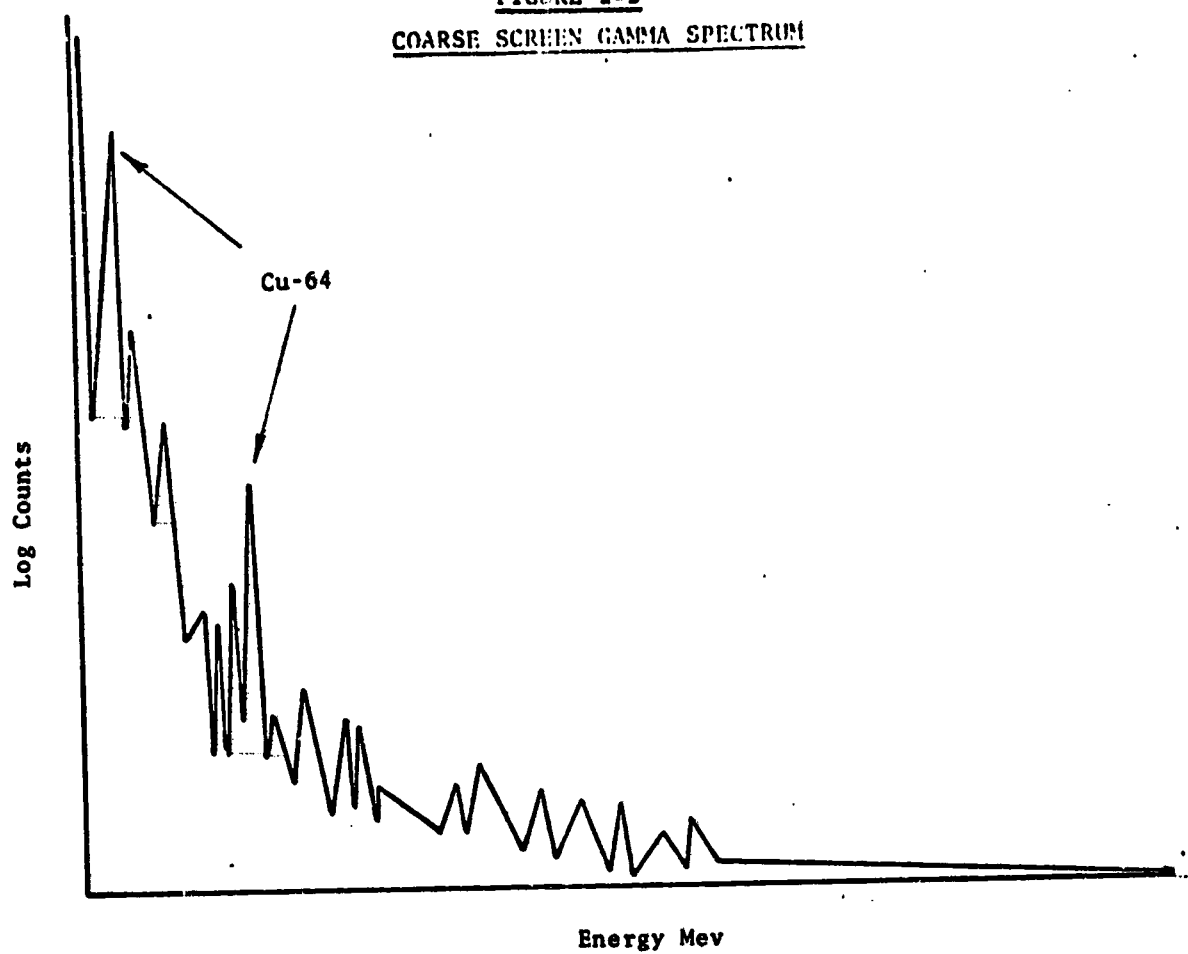
FIGURE 2-3

COARSE SCREEN GAMMA SPECTRUM

FIGURE 2-4
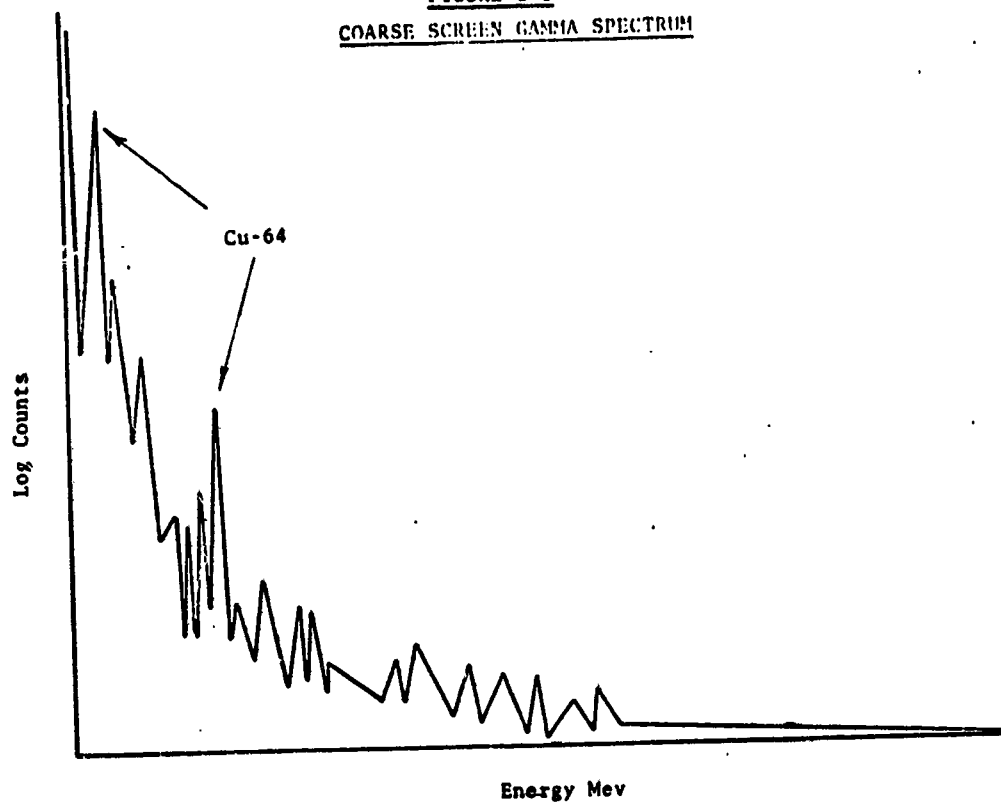
FINE SCREEN GAMMA SPECTRUM
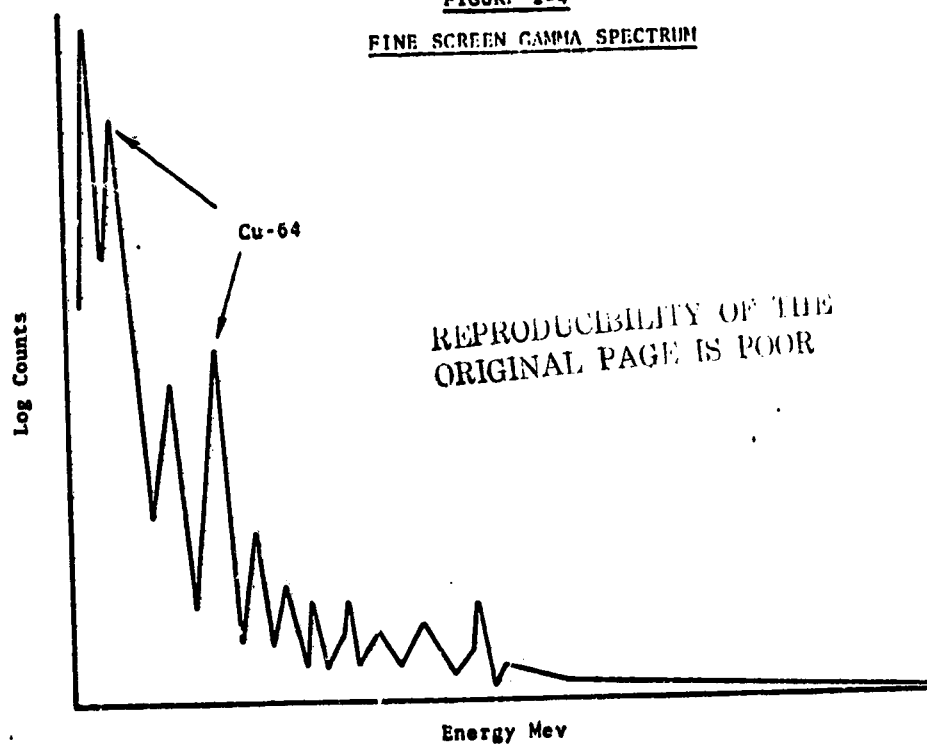
FIGURE 2-5

BACKGROUND GAMMA SPECTRUM

Log Counts

Energy Mev
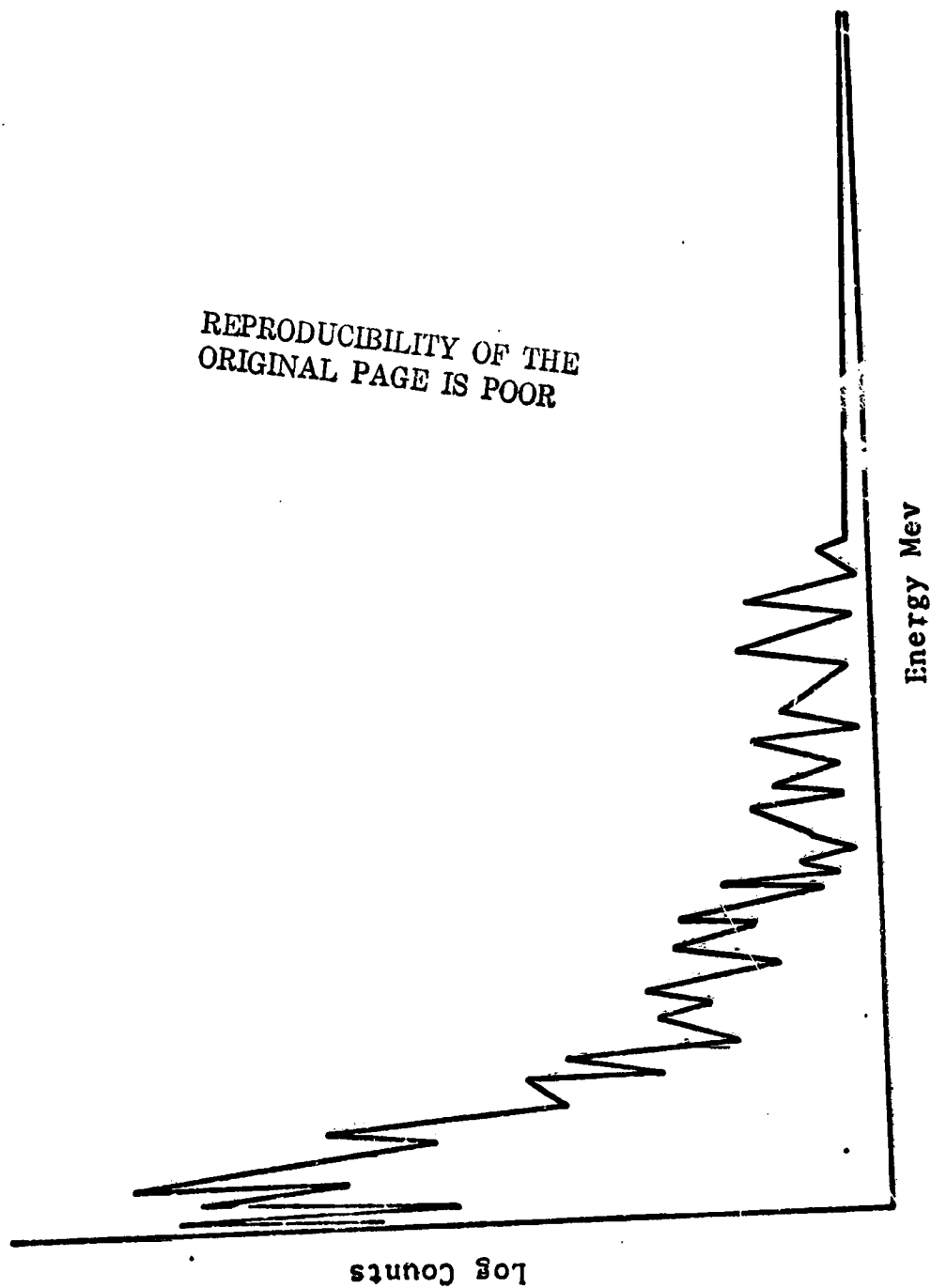
TEST NUMBER: 3

TEST ITEM: Solenoid Actuated Propellant Valve

2.1 Scope

The purpose of this test was to determine the contaminant generation characteristics of a Marquardt Valve, P/N T-12397 S/N 010, as a function of the number of cycles through which the valve has been operated. The test was designed to simulate certain typical operating conditions of flow and pressure. Some typical ambient conditions, i.e. temperature, fuel (water was used), acoustic noise level, and vibration, were not simulated. The test was conducted for the ma. mum design operating lifetime (20,000 cycles) of the valve. The valve was leak tested before, during, and after the tests. The contaminant generation was monitored, using auto-radiotracer techniques. The first 10,000 cycles used clean test fluid (water) while the second 10,000 cycles were conducted with fluid contaminated with AC Coarse Test Dust.

2.2 Procedure

The procedures followed in conducting the contaminant generation study are shown in the attached Test Plan. The valve components (spring, spring endcaps, poppet, and the valve seat and base) were irradiated for one hour at a flux of 1.5×10^{10} n/cm^2 - sec. This yielded a total activity of 9.5 mCi, almost all of which was Mn-56 from the steel components. The Fluorogreen seat, estimated at less than 1% of the total mass of material irradiated, yielded relatively little activity. Gamma spectra for the various valve components irradiated are shown in Figures 3-1 through 3-4. The activity calculation follows the Test Plan.

Following the irradiation and reassembly of the valve, the valve was installed in the test loop and tested as outlined in the Test Plan. A NaI scintillation detector system was used to monitor the three filters as the valve was cycled. Data was continuously recorded using a strip chart recorder. After about 6000 cycles, the test was temporarily stopped to relocate the valve in the test loop farther away from the three filter positions. This was done so that more shielding could be placed between the irradiated valve and the scintillation detector, thereby increasing the sensitivity of the detector to any activity buildup on the filters which might be occurring as the result of valve wear. Following the relocation of the valve, the test was resumed and carried out to completion according to the test plan.

2.3 Results and Analysis

Valve wear was detected when the filters were removed from the test loop and counted using a 3 inch NaI scintillation crystal and counting system. The filters were counted

TEST PLAN

1. Disassemble and clean the valve.

2. Identify the types of motion.

    a. Sliding - poppet, body, spring, and spring end caps.

    b. Impact - seat and base.

3. Identify the materials to be irradiated.

    a. Spring - Inconsl 700

    b. Armature - 446 CRES, electrolyzed Cr surface

    c. Seat - Fluorogreen (Glass-filled TFE)

4. Irradiate the valve components for 1 hr at 1 Kw (flux = 1.5×10^{10} n/cm^2-sec).

5. Clean and reassemble the valve.

6. Weigh filter screens and install them downstream of the valve (in this order):

    a. 80 x 700 square mesh or equivalent

    b. 325 x 2300 square mesh or equivalent

    c. Millipore pad (5 microns)

7. Install the valve in the test loop.

8. Perform an acceptance test:

    a. Run 20 cycles dry (no flow)

    b. Run 180 cycles with flow

    c. Flow conditions:

        (1) 180 psi back pressure with the valve closed.

        (2) 0.88 gpm (water) at 17 psi with the valve open.

9. Operate the valve through 10,000 cycles with flow (flow conditions as described for the acceptance test).

10. Remove the filters and replace them with similar, preweighed filters.

11. Count the first set of filters using a 3" NaI scintillation detector system.

12. Run 10,000 cycles with flow adding a total of 40 mg of AC Coarse Dust according to the following schedule:

    a. Add 2 mg and operate 500 cycles for a total of 10 adds (20 mg) and 5000 cycles.

    b. Add 4 mg and operate 500 cycles

        Totals: 24 mg and 5500 cycles

    c. Add 4 mg and operate 500 cycles

        Totals: 28 mg and 6000 cycles

    d. Add 2 mg and operate 1000 cycles

        Totals: 30 mg and 7000 cycles

e. Add 4 mg and operate 1000 cycles

　　　　Totals: 34 mg and 8000 cycles

f. Add 2 mg and operate 1000 cycles

　　　　Totals: 36 mg and 9000 cycles

g. Add 4 mg and operate 1000 cycles

　　　　Totals: 40 mg and 10,000 cycles

13. Terminate the test at 20,000 cycles; 10,000 without dust and 10,000 with dust additions. Leak test the valve.

14. Remove the second set of screens and count, using a 3" NaI scintillation detector system.

15. Weigh both sets of filters.

ACTIVATION OF VALVE COMPONENTS

$$A = \frac{m\sigma\phi Na}{MW} \ (1-e^{-0.693t/t_{\frac{1}{2}}}) \ (f) \times \frac{1mCi}{3.7 \times 10^7 \ dps}$$

A   =   Activity - mCi

m   =   Mass of sample irradiated - gm

σ =   Neutron absorbtion cross seciton - cm^2/atom

ϕ =   Neutron flux - n/cm^2 - sec

Na =   Avogadro's number - 0.6023×10^{24} atoms/mole

MW =   Molecular weight

$t_{\frac{1}{2}}$ =   Half life

f   =   Abundance of specific isotope

t   =   Irradiation time

Approximately 50 gm of stainless steel are to be irradiated. ^{56}Mn will be the only isotope produced to an appreciable extent,

$t_{\frac{1}{2}}$ =   2.58 hr

m   =   45 gm

f   =   0.02

MW =   55 gm/gmole

σ =   13 x 10^{-24} cm^2/atom

Irradiate the material for one hour at 1Kw:

t   =   1 hr

ϕ = 1.5×10^{10} n/cm^2-sec

Then:

$$A = \frac{(45)(13)(1.5 \times 10^{10})(0.6023)}{55(3.7 \times 10^7)} \ (1-e^{-0.693/2.58}) \ (0.02)$$

$$A = 5.20 \times 10^1 (1-e^{-0.268})$$

$$A = 5.20 \times 10^1 (1-0.765)$$

$$A = 12.2 \ mCi$$

in a heavy lead shield, thus minimizing the influence of the natural background radiation. The two sets of filters were counted within two hours of the respective parts of the test. These times corresponded to approximately 13 and 19 hours, respectively, from the time the parts had been irradiated. Gamma spectra for the six filters counted, background, and a calibration run are shown in Figures 3-5 through 3-12. Table 3-1 summarizes all the counts that were taken.

Analysis of the filters in this manner yielded the following results: the activity on the 325 x 2300 screen from the first part of the test was 820 cpm above background (Figure 3-8), the activity on the 5μ millipore pad was 554 cpm above background (Figure 3-5), and no activity was detected on the 80 x 700 screen from the first part of the test. These count rates are corrected for decay back to the time of removal from the reactor.

A 0.05μ Ci Cs-137 source was used for an approximate calibration of the counting system: Cs-137 having a gamma peak at 0.67 MeV, approximately the same energy as the Mn-56 peak at 0.84 MeV, and the two peaks being of approximately the same width. Using the derived calibration in terms of cpm/μCi and the initial specific activity of the irradiated parts, 10 mCi Mn-56/45gm, the following amounts of generated contaminant collected on the first set of filters were estimated (corrected for decay):

80 x 700 screen:	0-
325 x 2300 screen:	22.7μgm
Millipore pad:	15.4μgm

No activity was detected on any of the second set of filters. This could indicate that no wear occurred during the second part of the test. However, it would seem that addition of the AC coarse dust during the second part of the test would enhance the wear of the valve. Therefore, a more probable explanation is decay of the Mn-56 following the second part of the test. Due to decay of the Mn-56, the levels were too low to be detected. At the time the first set of filters was counted, the activity on the 325 x 2300 screen and the millipore pad was 23.3 and 17.7 cpm above background, respectively. The second set of filters was counted six hours, or about two half-lives of the Mn-56, after the first. Thus, assuming a buildup of generated contaminants on the second set of filters similar to that found on the first set, the activity level would be down by a factor of four, or only 4-5 cpm above background, which is not detectable with any degree of statistical reliability. Thus, while it is expected that some wear did occur during the second part of the test, the wear was too slight and the remaining activity level too low for the buildup of generated contaminants on the second

Because of the low levels of activity collected on the filters and the relatively high background radiation levels at the filter positions due to the proximity of the radiated valve, buildup of generated contaminants on the filters was not seen with the on-line detector. The relocation of the valve and the placement of additional shielding between the valve and the monitoring positions led to a significant reduction in the background level, but the overall system sensitivity was still such that the slight amount of contaminant buildup on the filters could not be seen on-line. Another factor which caused difficulty in monitoring the wear on-line was that the total activity was primarily from Mn-56, which decays with a relatively short half-life of 2.58 hr. Thus while it is expected that wear continued throughout the test, it became progressively more difficult to detect the activity buildup on the filters.

To verify the preliminary estimates of the weight of generated material collected on the screens, a small sample from the poppet was irradiated under conditions nearly identical to the initial irradiation. This provided an accurate calibration of the counting system. Using this calibration and the count rate data previously collected for the wear test, the amount of material collected on the filter was more accurately determined.

The small sample of material (32.6 mg) from the poppet of the valve was irradiated with neutrons using a nuclear reactor. The conditions of the irradiation very closely approximated the conditions of the initial valve irradiation. The irradiation parameters and the activation calculations are presented in the section "Activation Calculations" which follows. The activated sample was counted using a 3-inch NaI scintillation crystal and multi-channel analyzer counting system. The results were used as a standard to which the previously obtained results were compared in order to determine the amounts of generated contaminants collected on the filters.

The specific activity of Mn-56 in the irradiated prepared standard sample was 1.20×10^7 cpm/gm (at removal from the reactor). The following are the previously obtained activity levels (corrected for decay to the time of removal from the reactor) detected on the filters:

#1 - 325 x 2300 screen	8.20×10^2 cpm
#1 - Millipore pad	5.54×10^2 cpm

Using these data and the specific activity of the prepared standard, the following amounts of generated contaminants collected on the filters are calculated:

#1 - 325 x 2300 screen	27.8 micrograms
#1 - Millipore pad	18.7 micrograms

These values compare with the previously presented estimates of $22.7\mu gm$ and $15.4\mu gm$ for the screen and millipore pad, respectively. The differences between the values calculated here using the standard prepared from the valve poppet and those calculated earlier using the Cs-137 source are 20%. This difference is most likely due to slight deviations in the flux in either the first or both the first and second irradiations at the points of sample placement from the anticipated flux levels. In addition, the NaI crystal has a slightly different efficiency for counting photons depending on the energy of the emitted gamma ray. For the limited number of samples available, a reasonable estimate of the mass of generated contaminant collected on the filters is made by averaging the two values calculated for each filter:

$$\#1 - 325 \times 2300 \text{ screen} \qquad 25.2\mu gm$$
$$\#1 - \text{Millipore pad} \qquad 17.0\mu gm$$

A precise determination of the amounts of generated contaminant collected on the filters may be made by irradiating the standard sample under conditions identical to the conditions of the original valve irradiation. The critical parameter in obtaining results which are precisely comparable is the integrated neutron flux which must be the same for both irradiations. The first irradiation was performed at 1 Kw, and the second at 100 Kw; also, the beam port used in the first irradiation could not be used for the second irradiation therefore approximates, but does not duplicate, that of the first irradiation.

The power of the activation analysis techniques for quantitatively detecting minute amounts of wear of spacecraft propulsion system components has been clearly demonstrated. Quantities on the order of 20 gm would be impossible to determine with conventional techniques. This is particularly true when one considers the strong possibility of contaminants from the fluid, tubing, joints, and other parts of the test loop being deposited on the filters.

3.0 Conclusions

This test demonstrates the feasibility of using radioactively tagged components to measure wear. Even though the wear that took place in this short duration test was extremely small (about 38.1 micrograms were detected), the method was sensitive enough to detect this trace quantity. In subsequent tests other techniques will be tested for extending sensitivity of "on-line" monitors.

It is interesting to note that the pre-weighed screen sampler showed a "pickup" of 0.004 grams of contaminant after the initial 10,000 cycles of operation in the clean system. Apparently, the material was shaken from the system during cycling as the detectors show-

ed only 0.000038 grams of material released directly attributable to the valve. Yet under normal methods of testing the erroneous conclusion could be drawn that the 0.004 grams of material had come from the valve.

Similar results obtained after weighing the filters at the conclusion of the contamination cycle test showed a collection of 0.0392 grams (0.004 grams of contaminant were added to the system), yet the detectors indicated that none of the material originated in the valve.

The measurement of particle generation by components using nuclear tracer techniques appears to be quite feasible and appropriate even under extremely contaminated system conditions.

ACTIVATION CALCULATIONS

Activation of Marquardt Valve Poppet Sample

$$A(\mu Ci) = \frac{mf_1f_2N_a\sigma\phi}{MW}(1-e^{-0.693t/t_{1/2}}) \times \frac{1\mu Ci\cdot sec}{3.7\times10^4}$$

A = activity (μCi)
m = mass of sample (gm)
f_1 = fractional abundance of initial isotope for element
f_2 = weight fraction of initial element in the alloy
N_a = Avogadro's number (atoms/gmole)
σ = neutron absorption cross section (cm²/atom)
φ = flux (n/cm² sec)
MW = atomic weight of initial isotope (gm/gmole)
t = irradiation time (hr)
$t_{1/2}$ = half-life (hr)

For this activation,
m = 3.26×10^{-2} gm
f_1 = 1.00
f_2 = 2×10^{-2}
N_a = 0.6023×10^{24} atoms/gmole
σ = 13×10^{-24} cm²
φ = 5×10^9 n/cm²-sec
MW = 55 gm/gmole
t = 1 hr
$t_{1/2}$ = 2.58 hr

$$A = \frac{(3.26\times10^{-2})(1.00)(2\times10^{-2})(0.6023\times10^{24})(5\times10^9)}{(55)(3.7\times10^4)}(1-e^{-0.693/2.58})$$

$$A = 1.25 \times 10^1 (1-0.765) = 2.95 \ \mu Ci \rightarrow 0.90 \times 10^2 \ \frac{\mu Ci}{gm}$$

Measured count rate (corrected for decay) = 3.90×10^5 cpm
Weight of sample = 3.26×10^{-2} gm

$$\text{Specific activity} = \frac{3.90 \times 10^5}{3.26 \times 10^{-2}} = 1.20 \times 10^7 \frac{cpm}{gm}$$

For the material collected on the test filters, the specific activity of the Mn-56 is:

$$\frac{10^4 \mu Ci}{45gm} = 2.22 \times 10^2 \frac{\mu Ci}{gm}$$

$$\frac{\text{Specific activity of screen material}}{\text{Specific activity of standard material}} = \frac{2.22 \times 10^2}{0.90 \times 10^2} = 2.47$$

To determine the mass of material collected on the filters using the prepared standard, a correction factor of 1/2.47 = 0.405 must therefore be included in the calculations.

On December 3, at 10:34 P.M., Ch 85 ± 10 channels = 11,169 cpm

Removed from the reactor at 9:30 A.M., τ = 13 hr elapsed time

$$e^{\lambda t} = e^{0.693(13)/2.58} = 34.9$$

Therefore, the count rate at t = 0, is $1.117\times10^4 \times 3.49\times10^1 = 3.90 \times 10^5$ cpm

$$\left.\begin{array}{l}\text{Activity} = 5.90 \ \mu Ci \\ \text{Count rate} = 3.90 \times 10^5 \ cpm \\ \text{Weight of standard} = 3.26 \times 10^{-2} gm\end{array}\right\} \rightarrow 1.20 \times 10^7$$

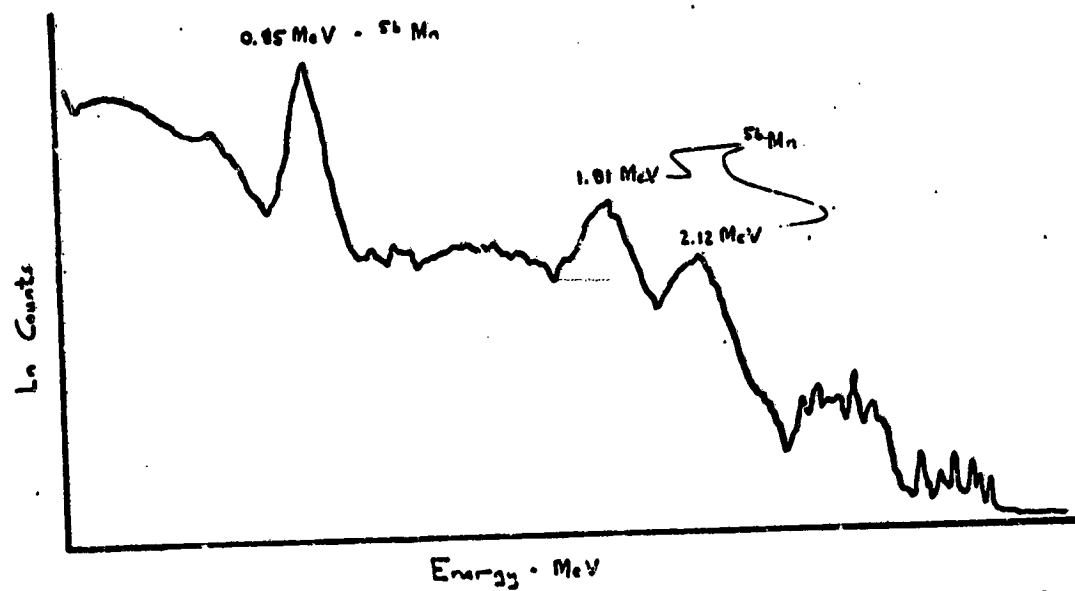325 x 2300 screen: $\dfrac{8.20 \times 10^2 \ cpm}{1.20 \times 10^7 \ \frac{cpm}{gm}} \times 0.405 = 27.8$ μgm

Millipore pad: $\dfrac{5.54 \times 10^2 \ cpm}{1.20 \times 10^7 \ \frac{cpm}{gm}} \times 0.405 = 18.7$ μgm

TABLE 3-1

SUMMARY OF GAMMA SPECTRA

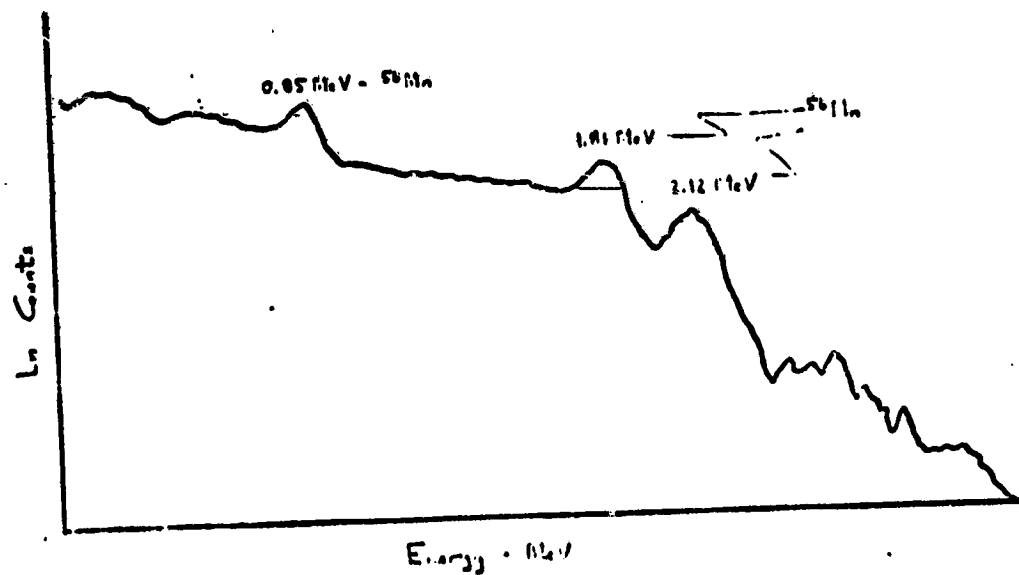| Figure No.		Counting Time (minutes)
C-5	Spring end cap	---
C-6	Valve seat and base	---
C-7	Poppet	---
C-8	Spring	---
C-9	Background	10m
C-10	#1 Millipore pad	10m
C-11	#1 80 x 700 screen	10m
C-12	#1 325 x 2300 screen	10m
C-13	#2 80 x 700 screen	10m
C-14	#2 325 x 2300 screen	10m
C-15	#2 Millipore pad	10m
C-16	0.05 µCi Cs-137 source	1m

FIGURE 3-1

SPRING END CAP

FIGURE 3-1 — SPRING END CAP

FIGURE 3-2

VALVE SEAT AND BASE

FIGURE 3-2 — VALVE SEAT AND BASE

FIGURE 3-3

POPPET

FIGURE 3-4

SPRING

FIGURE 3-5

BACKGROUND
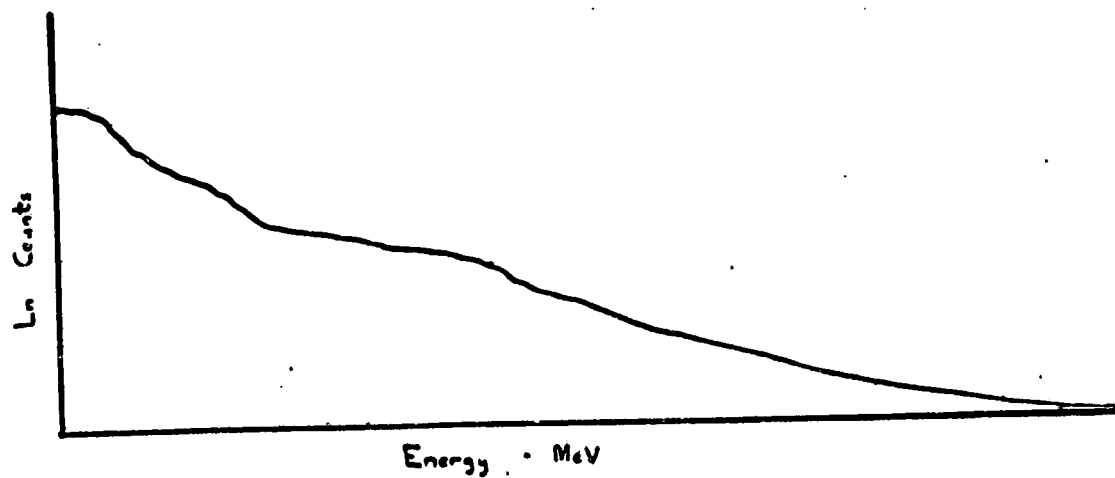
FIGURE 3-6
#1 - 5 μ MILLIPORE FILTER, UNCONTAMINATED RUN

FIGURE 3-7

#1 80 x 00 SCREEN

Energy - MeV
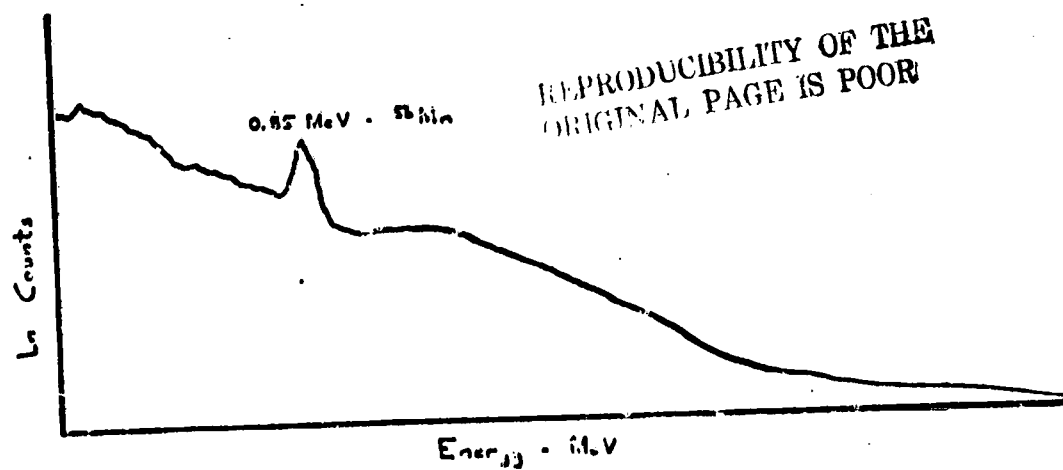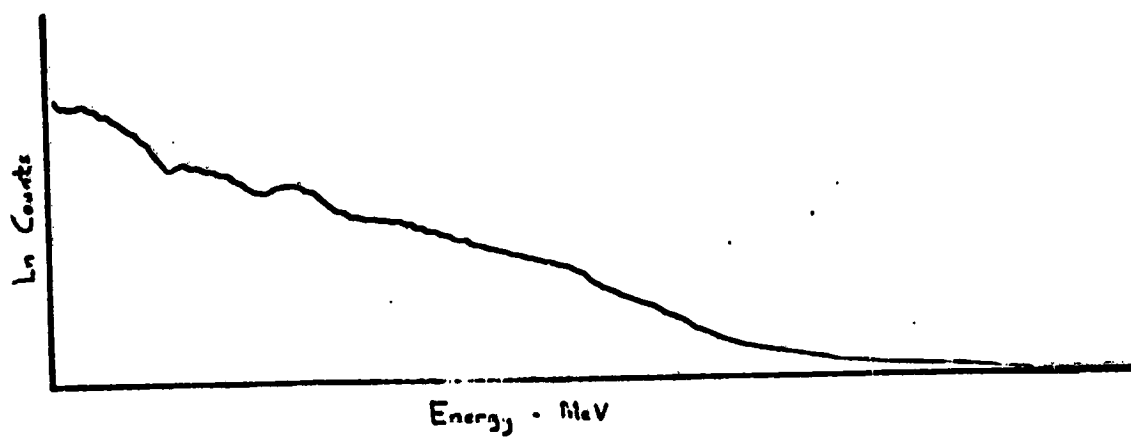
REPRODUCIBILITY OF THE
ORIGINAL PAGE IS POOR

FIGURE 3-8

#1 325 x 2300 SCREEN
UNCONTAMINATED RUN

0.85 MeV - 54-Mn

Energy - MeV

63
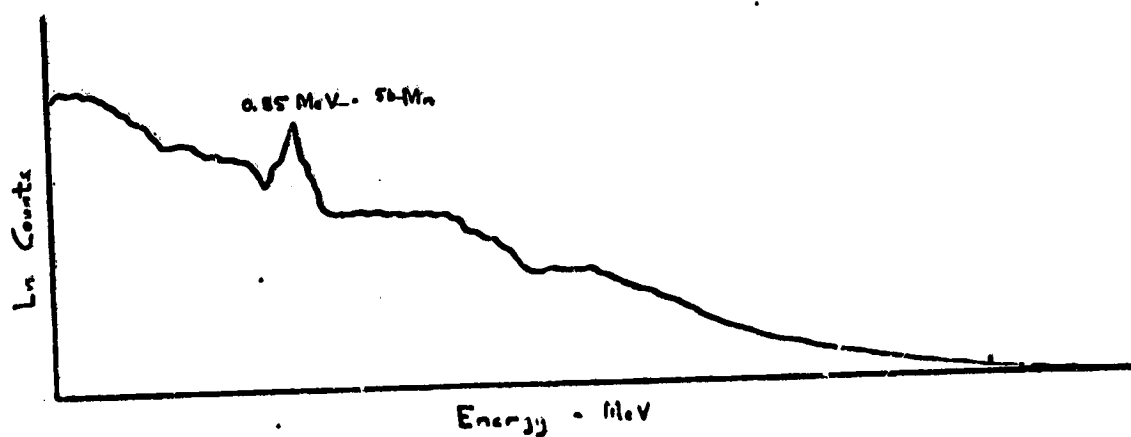
FIGURE 3-9
#2 80 x 700 SCREEN
CONTAMINATED RUN
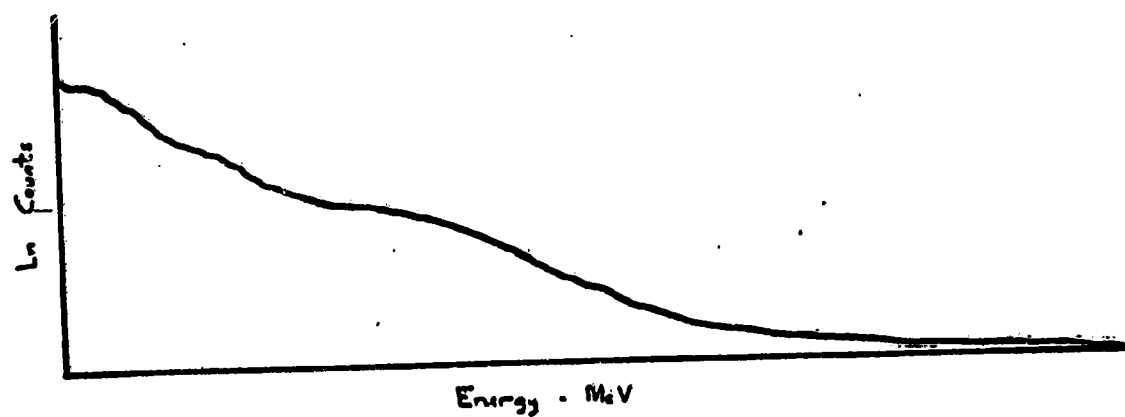
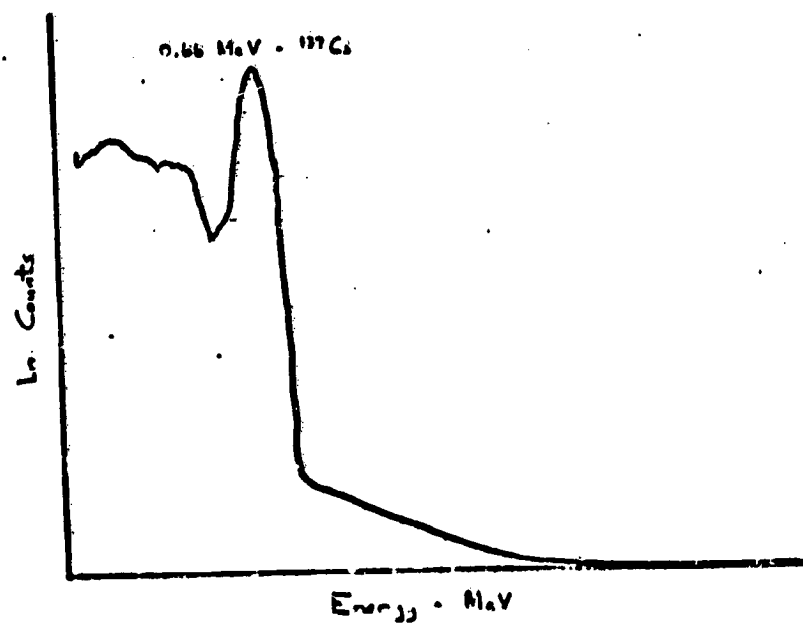FIGURE 3-10
#2 325 x 2300 SCREEN
CONTAMINATED RUN

FIGURE 3-11

#2 - S / MILLIPORE FILTER, CONTAMINATED RUN

Energy · MeV

FIGURE 3-12

0.05 µCi Cs-137 SOURCE

0.66 MeV - 137Cs

Energy · MeV

TEST NUMBER: 4

TEST ITEM: Hydraulic Actuator

1.0 Scope

The purpose of this test was to determine the contaminant generation characteristics of a typical hydraulic actuator (as shown in Figure 4-1), as a function of the number of cycles through which the actuator has been operated. The unit was operated under unloaded and loaded operating conditions through a total of 5,400 cycles. Leakage from the actuator was measured intermittently during the test. The contaminant generation was monitored using auto-radiotracer techniques.

2.0 Procedure

The procedures followed in conducting the wear study are shown in the Test Plan. The actuator components (body, piston, and bushing) were irradiated for 30 minutes at a flux of approximately 5×10^{10} n/cm^2-sec. This yielded a total activity of approximately 10 mCi, mainly Cu-64 in the bushing and Cu-64 and Mn-56 in the actuator body.

Samples of material removed from the 7075 aluminum body and Cu-Be bushing were irradiated along with the actuator components. These samples were used as the calibration standards. The piston, being made of 440C stainless steel, is much harder than either the body or the bushing. It was therefore assumed that if any particle generation were to occur it would be from the body and bushing rather than from the piston. This assumption was later verified by the absence of the characteristic gamma decay peaks corresponding to the alloying constituents of 440C stainless steel in the gamma spectra of the generated contaminants.

Following the irradiation and reassembly of the actuator, it was installed in the test loop and tested as outlined in the Test Plan. A schematic diagram of the test loop is shown in Figure 4-2. One modification to the Test Plan was made during the test. After 2700 cycles, the differential pressure across the screens built up to a higher value than anticipated. The screens were removed from the test loop and replaced with clean screens. The test was then carried out as planned to completion; i.e., to a total of 3,600 cycles unloaded (2,700 cycles with the first set of screens and 900 cycles with the second set) and 1,800 cycles with the actuator loaded (using a third set of screens). The activity collected on the screens was intermittently monitored during the run using a two-inch NaI scintillation detector.

3.0 Test Plan - Hydraulic Actuator

1. Install the hydraulic actuator in the test loop and install the following filters

downstream of the actuator:

   a. 80 x 700 screen   (40 GBR)
   b. 325 x 2300 screen (10 GBR)

2. Test conditions:

   a. Fluid: MIL-H-5606
   b. Pressure Source: Greer supply unit
   c. Pressure: 3000 psi
   d. Line size: 1/4 inch
   e. Flow rate: approximtely 0.45 gpm
   f. Maintain a temperature record and use the provided heat exchanger as necessary.

3. Install a 325 x 2300 screen in the pressure line.

4. Cycle the 4-way solenoid valve to pressurize and exhaust the cylinder. The valve is to operate at 3000 psi on the inlet and downstream sides. A snubber or needle valve is to be used to restrict the travel time and impact load of the actuator.

5. Clamp the cylinder near the ends to avoid restriction of the bore.

6. Measure the leakage across the actuator prior to the test by applying 3000 psi to the retraction port and measuring the effluent from the extension port.

7. Cycle the actuator through 3,600 cycles at a cycling frequency of 30 cycle per minute, with the actuator in the unloaded condition.

8. Pass the effluent from the actuator under both travel directions through the collection screens. Monitor the activity of the collection screens with a portable detector.

9. Repeat the leakage test (as in Step 6).

10. Remove the replace the screens.

11. Cycle the actuator through 1500 cycles at 30 cycles per minute with a 2 pound weight attached to the rod end eye bolt to simulate side loading conditions.

12. Repeat Step 8.

13. Repeat the leakage test (Step 6).

14. Remove the screens for the off-line counting and analysis.

4.0 Results and Analysis

The gamma spectra for the actuator body and bushing calibration samples are shown in Figures 4-3 and 4-4 respectively. The body sample yielded a Mn-56 peak of 2.81×10^2 cpm/mg and a Cu-64 peak of 3.97 x 10^2 cpm/mg; the bushing sample yielded a Cu-64 peak of 7.77×10^3 cpm/mg. In analyzing these spectra, and all spectra which follow, the count rate considered is the total number of counts in the peak channel and five channels on either side of the peak divided by the counting period. The calibration samples are normalized to activity per milligram and are subsequently used to calculate the mass of generated contaminant collected on the various screens used during the test.

The basic procedures used to analyze the test data are as follows:

   a. Count each screen using a 3" NaI scintillation detector and counting system, and determine the Mn-56 and Cu-64 activity on each screen.

b. Using the Cu-64 calibration factor for the body, calculate the Cu-64 activity corresponding to the measured Mn-56 activity. The Mn-56 can come only from the actuator body. Subtracting the corresponding Cu-64 activity from the total Cu-64 activity on the screen will yield the Cu-64 activity due to the bushing.

c. From the measured and calculated activities from steps (a) and (b) corrected for decay back to the time of the counting of the standards, using the calculated calibration factors, calculate the mass of generated contaminant from the actuator body and bushing collected on each of the filters.

The gamma spectra for the six screens used during the test are shown in Figures 4-5 through 4-10. From these figures, it is seen that the gamma spectrum for the #1 Fine Screen is the only one which shows a definite Mn-56 peak. Using the data from the counting of this screen and the outlined analysis procedures, the masses of contaminant generated from the body and bushing were calculated: the values are 0.42 mg and 0.19 mg, respectively.

At this point it is assumed that the ratio of body material to bushing material remained constant throughout the test. By this assumption, there should be a Mn-56 peak in the spectrum for each of the screens, but, as already indicated, these peaks do not occur. Close inspection of the data reveals some activity, though not a discernable peak, at the Mn=56 energy of 0.84 MeV. The absence of the Mn-56 peaks is possibly explained by the following two considerations:

a. The total activity on each of the remaining screens ranges from approximately 1 to 10% of the total activity on the #1 Fine Screen. The Mn-56 activity is therefore also reduced by a similar factor.

b. The #1 Fine Screen was one of the first screens counted; the others were counted at times from 3 to 5 hours later, or 1 to 2 half-lives of Mn-56. This also resulted in a significant reduction in the already low Mn-56 activity level.

Accepting the validity of this assumption enables one to distinguish between Cu-64 activity on the remaining five screens due to particle release from the actuator body and that from the bushing. By the assumption, the ratio of body Cu-64 activity to bushing Cu-64 activity will be constant throughout the test and equal to that calculated for the #1 Fine Screen. Then, using the separated Cu-64 activities and the appropriate calibration factors, the masses of contaminants released from the two parts and collected on the screens can be calculated. The results are shown in Table 4-1.

The results indicate a total of 0.69 mg of generated contaminant collected on the filters throughout the test. The result for the coarse screen with actuator load d, while insignificant compared to the 0.69 mg of total collected contaminant, is quite significant in a different respect. For that particular case, 0.98×10^{-3} mg, or less than 1 microgram of material was detected.
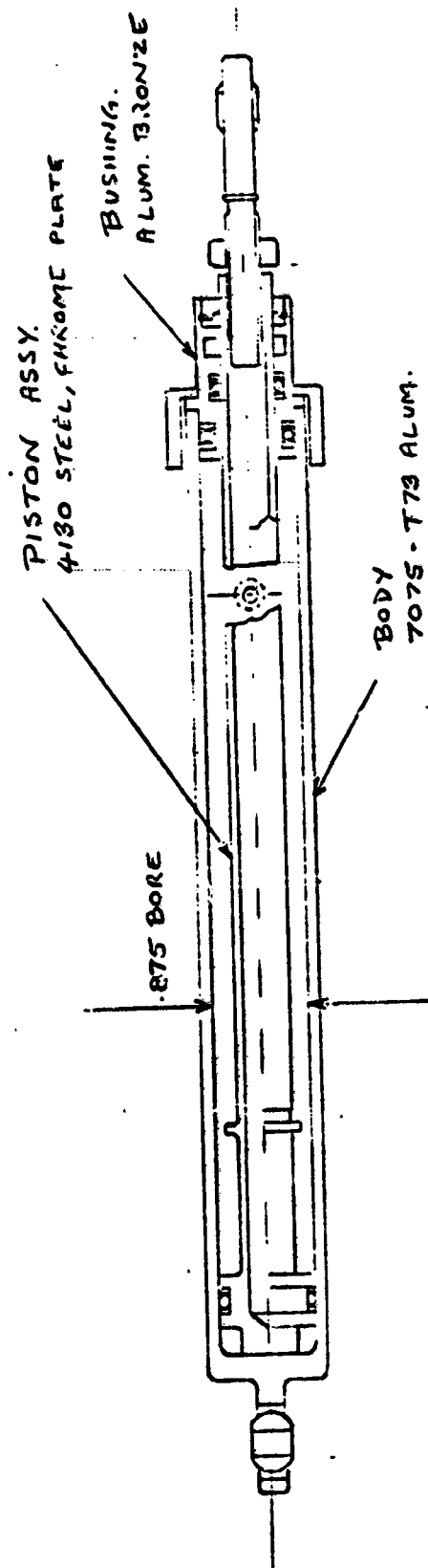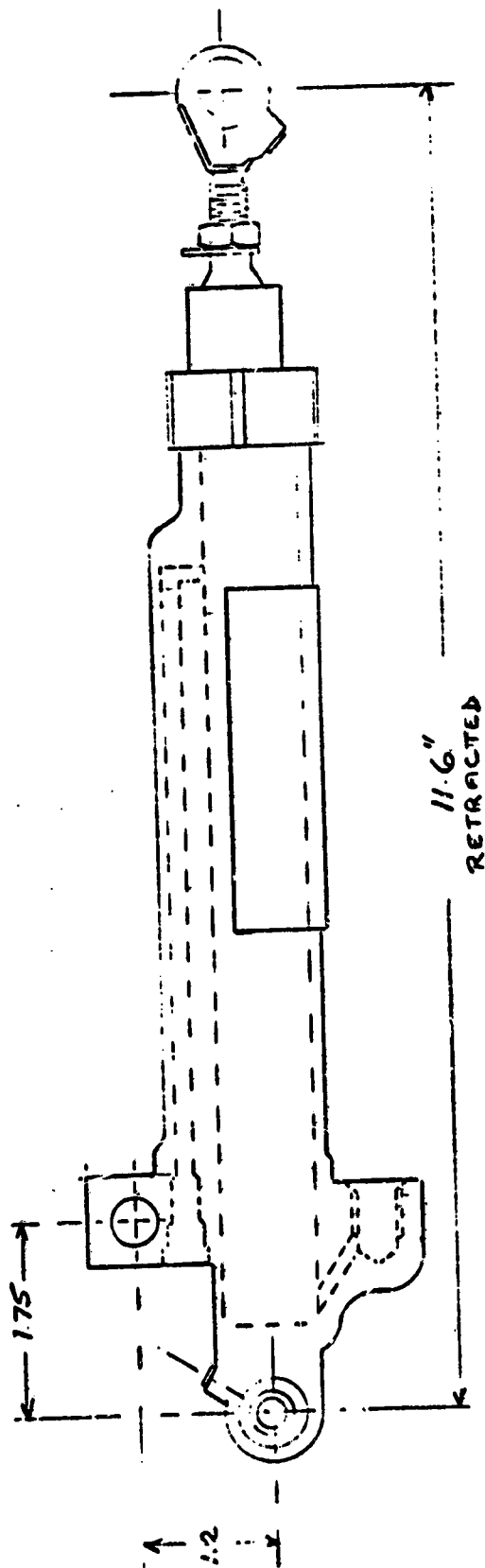
Figure 4-11 shows the contaminant generation history of the actuator as a function of the number of cycles through which it has been operated. While the number of data points is somewhat limited, the curves do illustrate several significant features. For either body or the bushing, the amount of contaminant collected on the coarse screens is a small fraction (approximately 2%) of that collected on the fine screens. Therefore, Figure 4-11(b) very nearly approximates the total contaminant generation curves for the two parts. The curves of Figure 4-11(b) show a sharp initial rate of generation which is characteristic of a breaking in period. This is followed by a leveling off of the generation rate, which is also shown. The side-load was added at 3,600 cycles. The curves shown that this added load did not have the effect increasing the rate of particle generation. The fact that the amount of generated contaminant collected on the coarse screens was generally only approximately 2% of that collected on the fine screen gives a good indication as to the particle size distribution of the generated contaminant. The particles are primarily between 10 and 40 microns in size.

5.0 Conclusions

The results of this test clearly indicate two of the principle advantages of the auto-radiotracer techniques used in the contaminant generation study. First, if the constituent materials of the component parts are such that they will yield different isotopes upon irradiation, these different isotopes can be used to quantitatively measure the overall particle generation of the component during operation, and can also be used to distinguish between sources within the component. With this in mind, parts could be doped with small quantities of materials during fabrication so as to serve as radiotracers upon irradiation. Second, the results indicate the high sensitivity of the auto-radiotracer technique as applied to contaminant generation studies. In this test, quantities of generated contaminant of less than one microgram were easily detected.

TABLE 4-1

MASSES OF GENERATED CONTAMINANT COLLECTED
HYDRAULIC ACTUATOR TEST

Actuator Part and Loading Conditions	Mass of Collected Generated Contaminant (milligrams)		
	Coarse Screen	Fine Screen.	Total
1. Body, unloaded	9.60×10^{-3}	4.49×10^{-1}	4.59×10^{-1}
2. Body, loaded	2.14×10^{-3}	1.42×10^{-2}	1.63×10^{-2}
3. Body, total	1.17×10^{-2}	4.63×10^{-1}	4.75×10^{-1}
4. Bushing, unloaded	4.33×10^{-3}	2.03×10^{-1}	2.07×10^{-1}
5. Bushing, loaded	0.98×10^{-3}	6.45×10^{-3}	7.43×10^{-3}
6. Bushing, total	5.31×10^{-3}	2.09×10^{-1}	2.14×10^{-1}
7. Total generated contaminant	1.70×10^{-2}	6.72×10^{-1}	6.89×10^{-1}

FIGURE 4-1

HYDRAULIC ACTUATOR ASSEMBLY

The following labels appear on the figure:

- 11.6" RETRACTED
- 1.75
- .2
- PISTON ASSY. 4130 STEEL, CHROME PLATE
- BUSHING. ALUM. BRONZE
- BODY 7075-T73 ALUM.
- .875 BORE

FIGURE 4-2

SCHEMATIC DIAGRAM OF THE HYDRAULIC ACTUATOR TEST LOOP
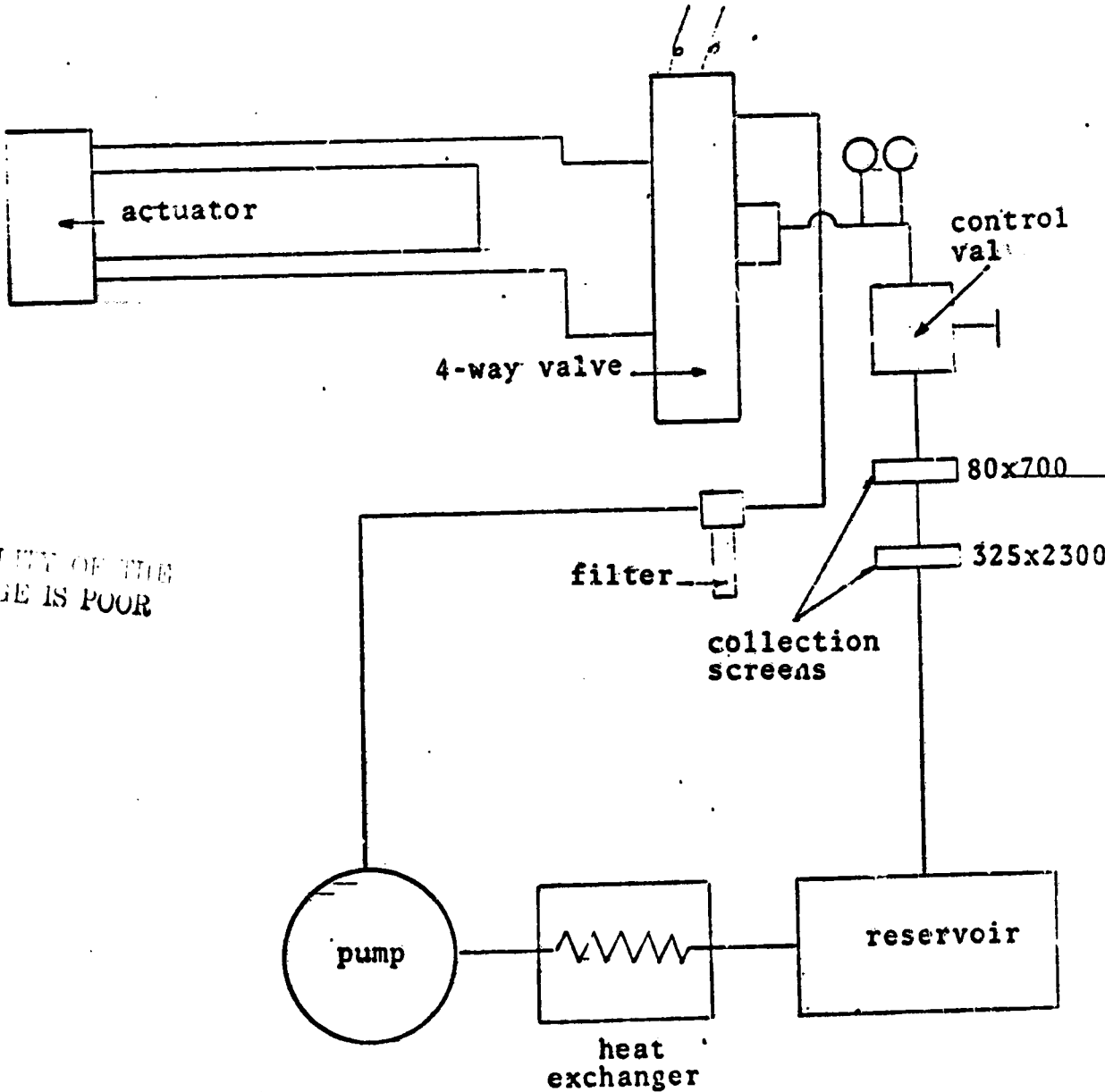
FIGURE 4-3

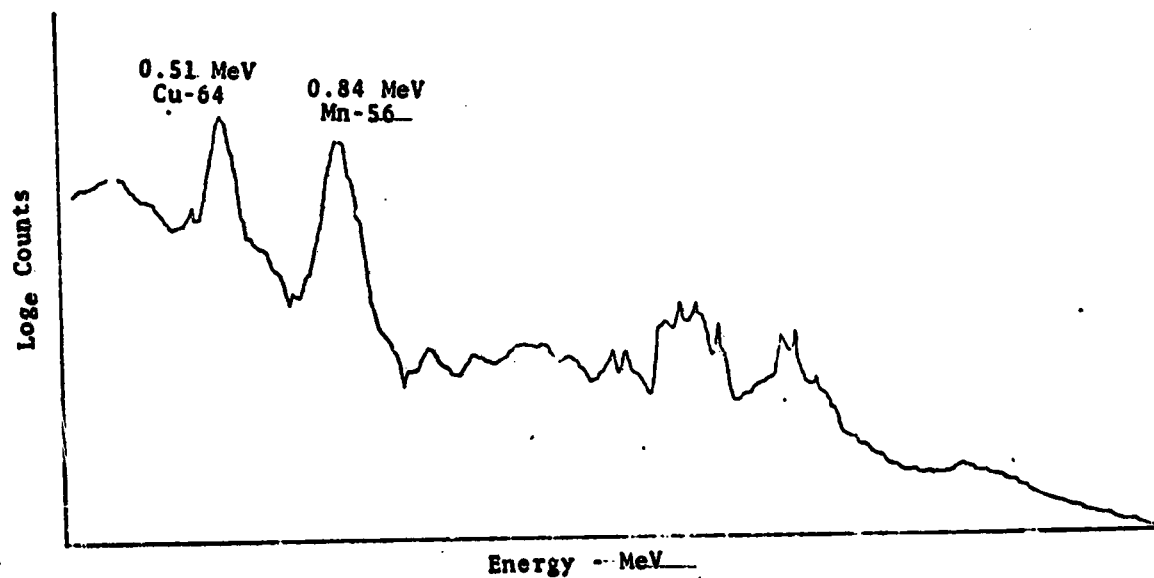GAMMA SPECTRUM OF THE ACTUATOR BODY, CALIBRATION SAMPLE

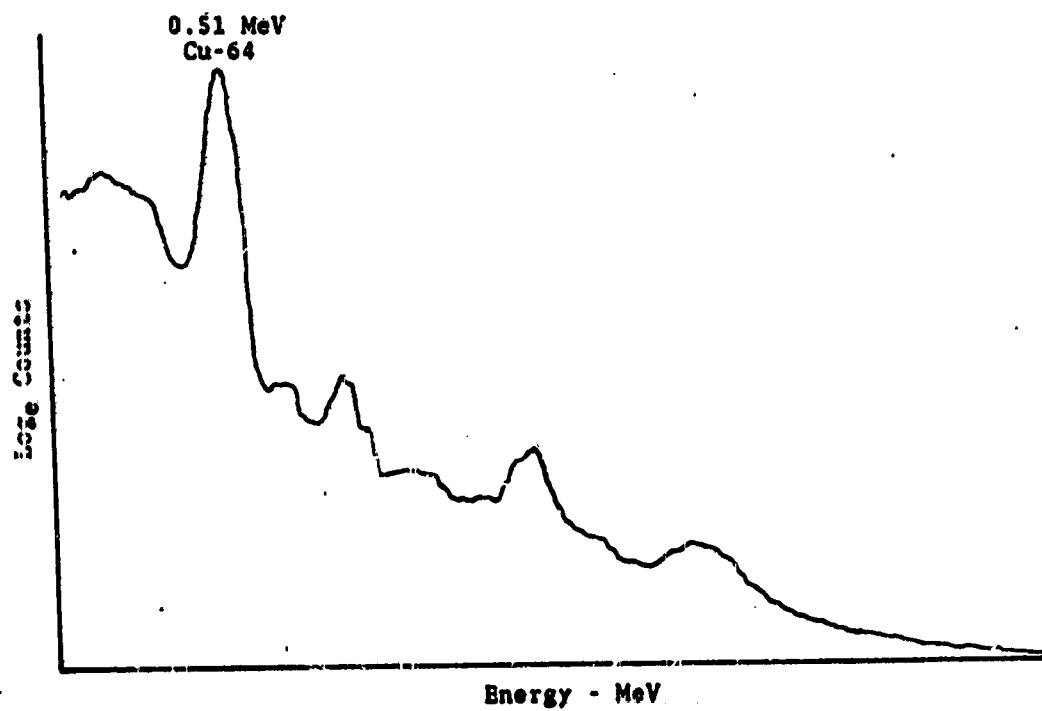FIGURE 4-4

GAMMA SPECTRUM OF THE BUSHING CALIBRATION SAMPLE

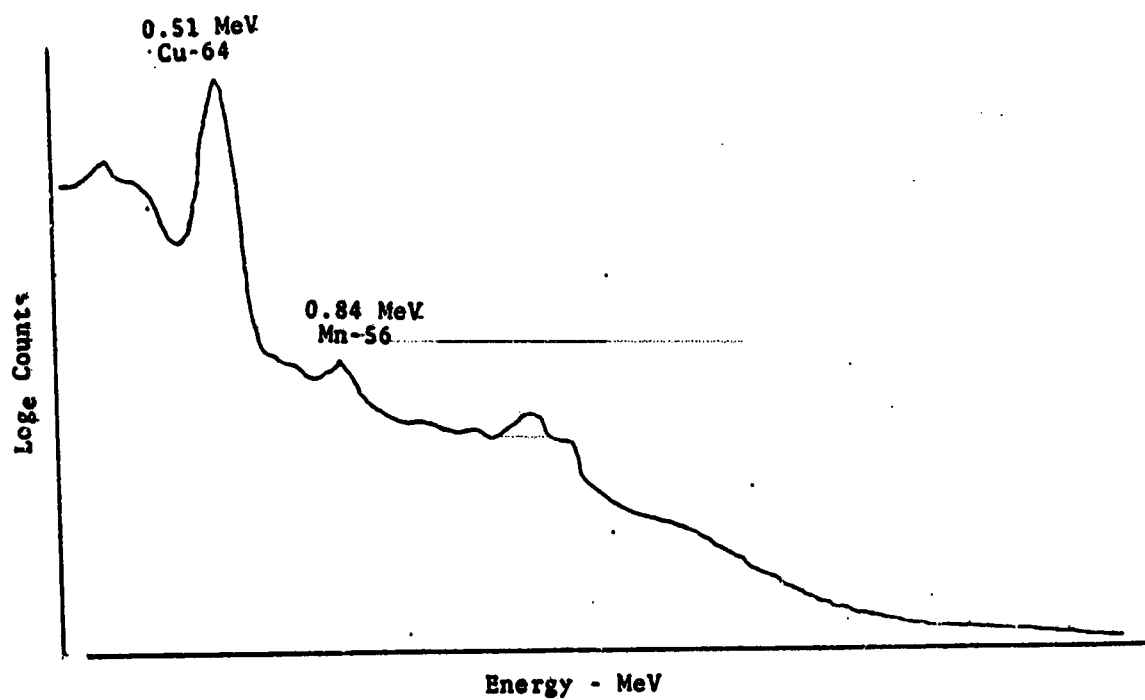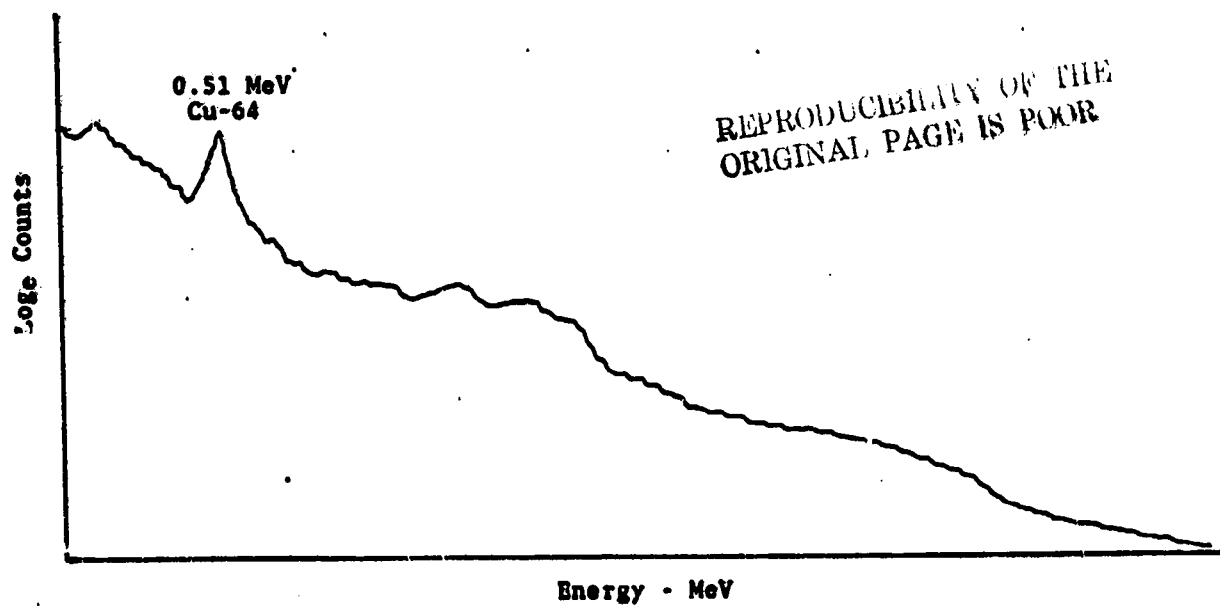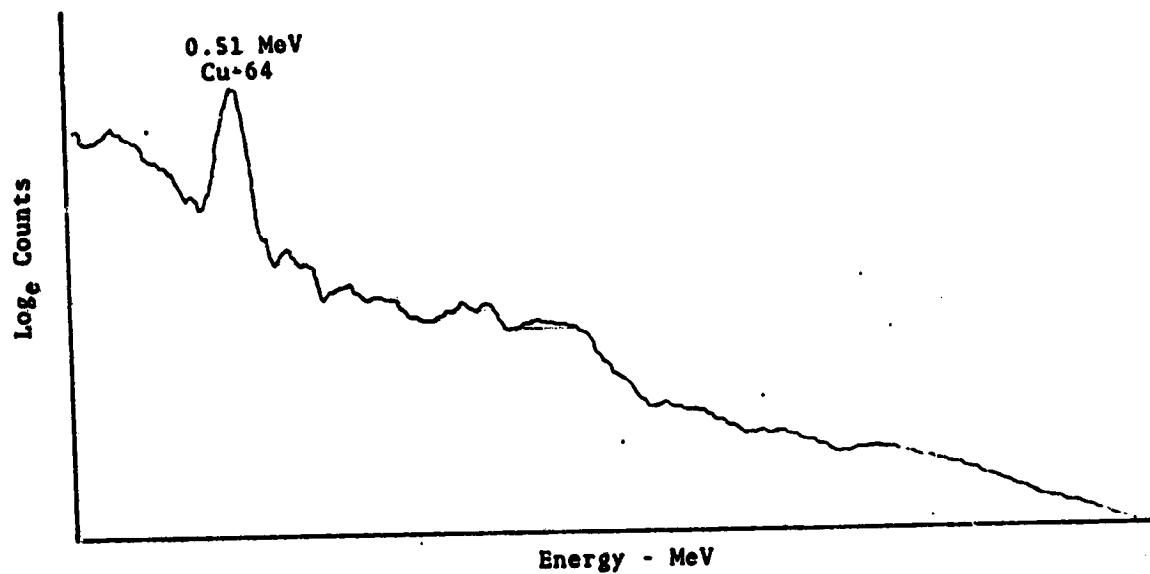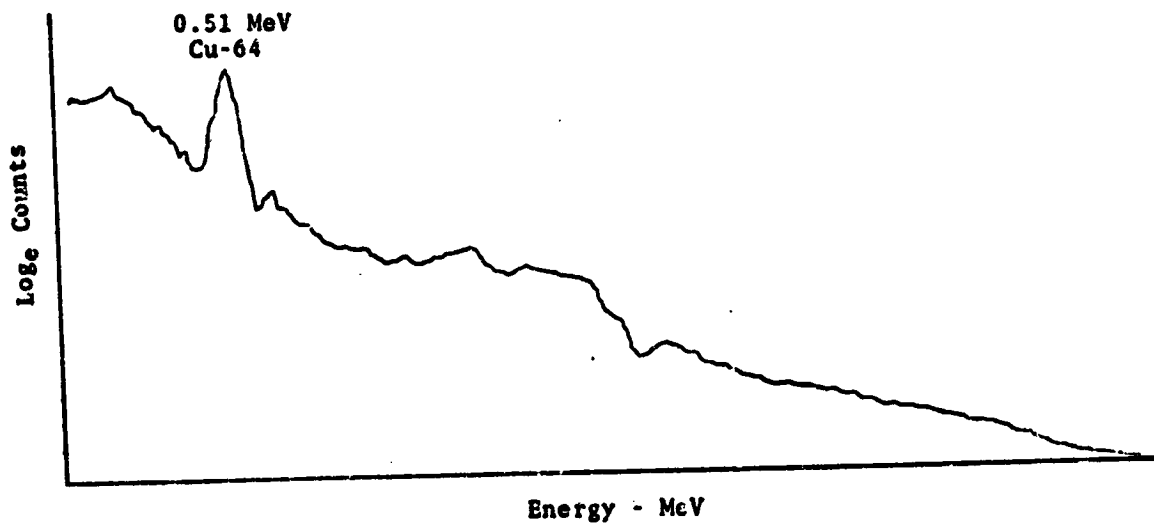FIGURE 4-5

GAMMA SPECTRUM OF THE #1 FINE SCREEN

(0-2700 cycles)

FIGURE 4-6
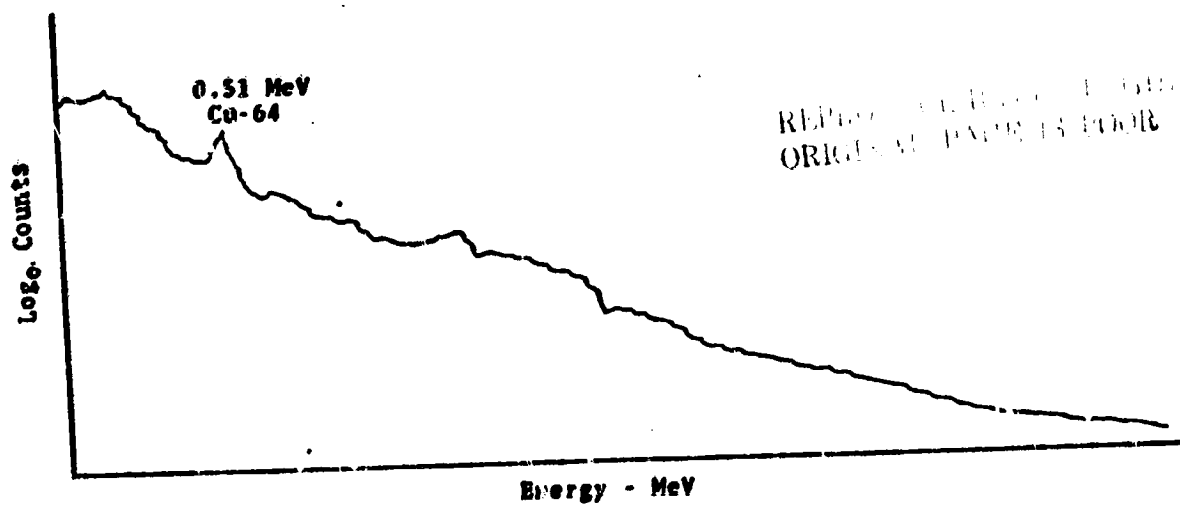
GAMMA SPECTRUM OF THE #1 COARSE SCREEN

(0-2700 cycles)

FIGURE 4-7
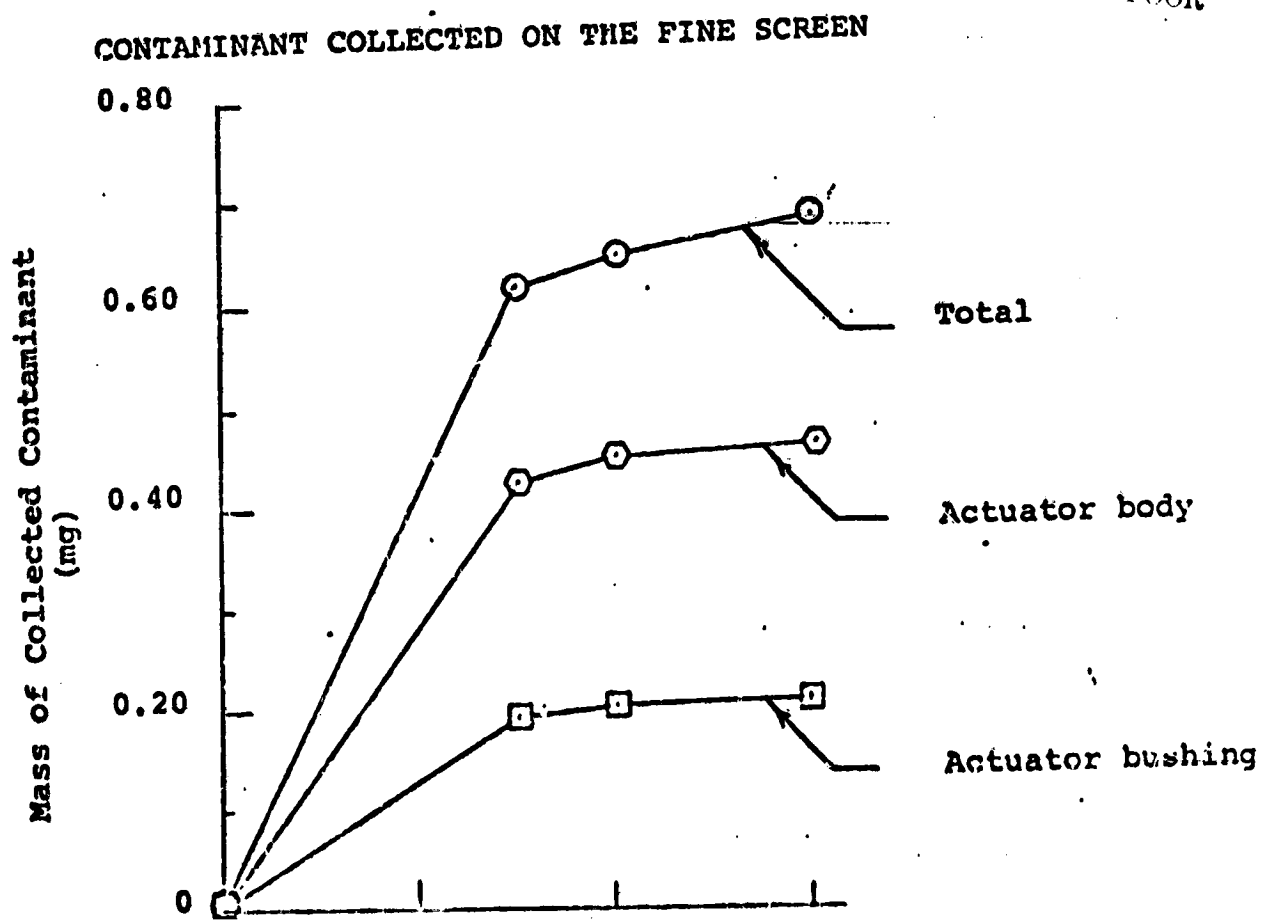
GAMMA SPECTRUM OF THE #2 FINE SCREEN

(2700-3600 cycles)

0.51 MeV
Cu-64

Log$_e$ Counts

Energy - MeV

FIGURE 4-8

GAMMA SPECTRUM OF THE #2 COARSE SCREEN

(2700-3600 cycles)

0.51 MeV
Cu-64

Log$_e$ Counts

Energy - MeV

75
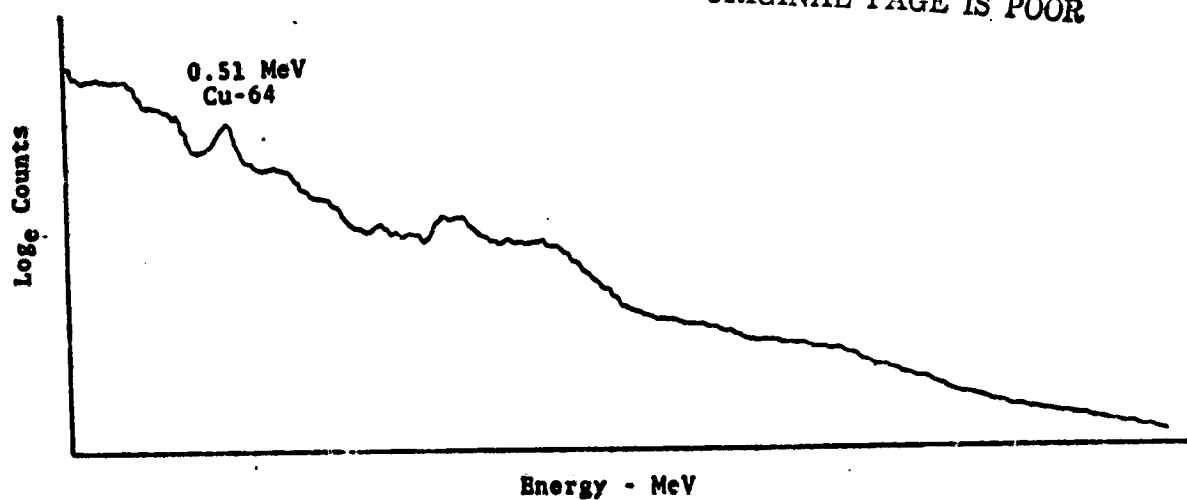
GAMMA SPECTRUM OF THE #3 FINE SCREEN

(3600-5400 cycles)

0.51 MeV
Cu-64

Log$_e$ Counts

Energy - MeV

FIGURE 4-10

GAMMA SPECTRUM OF THE #5 COARSE SCREEN

(3600-5400 cycles)

0.51 MeV
Cu-64

Log$_e$ Counts

Energy - MeV

FIGURE 4-11: CONTAMINANT GENERATION HISTORY OF THE HYDRAULIC ACTUATOR

CONTAMINANT COLLECTED ON THE COARSE SCREEN

CONTAMINANT COLLECTED ON THE FINE SCREEN
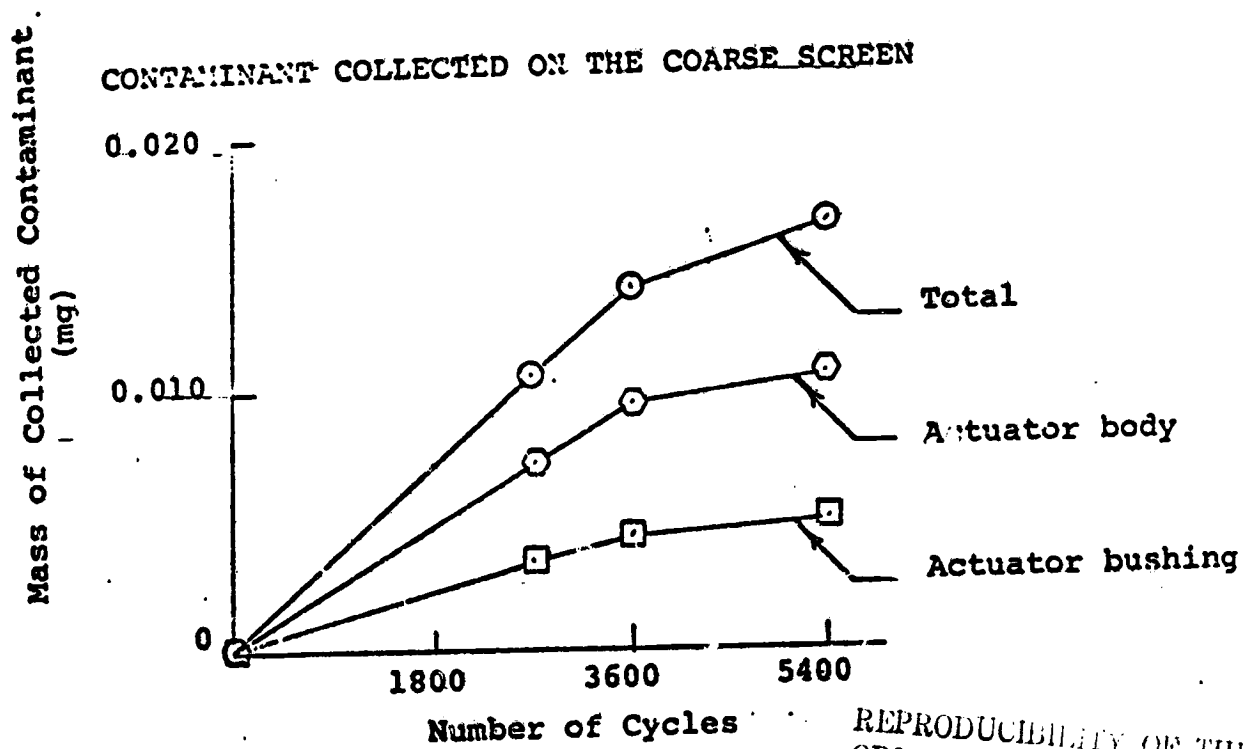
TEST NUMBER: 5

TEST ITEM: Ball Bearing Assembly

1.0 Scope

The purpose of this test was to determine the contaminant generation characteristics of a typical high speed ball bearing assembly while operating in a cryogenic liquid. A special bearing test apparatus designed for use with cryogenic liquids was provided by the Rocketdyne Division of North American Rockwell Corporation. It was originally planned to run the bearing for 3 hours or until failure using liquid nitrogen at 4 gpm and 225 psia inlet pressure. However, one of the two bearing assemblies installed in the tester failed after only 12 minutes or 240,000 revolutions and the test was concluded. During the run the generated contamination was collected on filter screens in the flow system, and these were monitored on-line using auto-radiotracer techniques. Bearing incipient failure was noted on the monitors by rapid accumulation of bearing material prior to audible indications of failure.

2.0 Procedure

The test procedures followed in conducting this study are shown in the Test Plan. A schematic diagram of the bearing tester and the test loop is shown in Figure 5-1. The shaft of the bearing tester and two bearings were irradiated for 1 hour at a flux of approximately 5×10^{10} n/cm^2 sec. This yielded a total activity of 5 mCi, mainly Cu-64 and Mn-56, which have half-lives of 12.8 hr. and 2.58 hr., respectively.

Following irradiation, the bearing tester was reassembled and installed in the test loop. The test was then run as outlined. A 2-inch NaI scintillation detector was placed near each of the two filter positions. Each detector had a separate readout. The output of the detector monitoring the fine screen was also connected to a strip-chart recorder and the relative activity of material collected on the fine screen was recorded continually during the test.

The tester was run briefly (3 minutes) prior to the beginning of the continual operation portion of the test. This was done primarily to check out the test loop. Following the system checkout, the bearing tester was brought to 20,000 rpm and run at that speed for the remainder of the test.

Following the test, the filters were removed from the test loop and counted using a 3-inch NaI scintillation detector and a multi-channel analyzer. From the results of these measurements, the mass of generated contaminants collected on the screens was determined.

3.0 Results and Analysis

During the early minutes of the continual operation portion of the test, a s early in-
crease in the activity level of each of the filters was detected. This is shown in
Figures 5-2 and 5-3 which represent the meter readings and a reproduction of the strip-
chart recording, respectively. After 12 minutes of continual operation, a sharp rise
in the activity levels was detected. This is also shown in Figures 5-2 and 5-3. Short-
ly following this rapid increase in collected activity, a marked increase in the high
frequency sound level originating from the bearing tester was noted. It was assumed
that these two effects had been caused by the failure of a component in the bearing
tester, and one minute later the test was terminated.

Following the test, the unit was disassembled and visually inspected. This insepction
indicated that the inner race of one of the bearings was binding.

Figures 5-4 and 5-5 show the gamma spectra for the two screens used in the test. Each
screen was counted twice and the spectra indicate the relative decay of the various
radioisotopes present in the screen. A small portion of the collected contaminant was
removed from one of the screens, counted, and weighed. Using this as a calibration
standard, the mass of generated contaminant collected on the screens was determined.
The results are 36.7 mg on the coarse screen and 44.8 mg on the fine screen. These
relatively large amounts of collected contaminant also confirm the component failure.

The two bearings used for these tests consisted of 440C stainless steel inner and outer
races and a K-Monel cage in one bearing and a Rulon-J cage in the other. Of these
materials, K-Monel is the only one in which copper is present. Therefore, the presence
of significant levels of Cu-64 on the filters, as illustrated by the gamma spectra,
indicates appreciable wear of the K-Monel cage. It was the bearing with the K-Monel
cage which was found, by visual inspection following the test, to have failed. Mangan-
ese, present in both K-Monel (1.5%) and 440C stainless steel (1.0%), was the source of
the Mn-56 detected on the screens.

The detection of Cu-64 activity on the screens when copper is present only in the K-Monel
cage provides positive indication that it was the K-Monel cage which yielded the major
portion of the collected contaminant and that it was this part of the bearing which in
all likelihood resulted in the component failure.

Figure 5-6 shows the bearing tester in operation. Liquid nitrogen from a supply to the
right of the photograph is flowing through the bearing tester on the table and thence
through the two filter screens in front to the gamma detectors in their cylindrical lead

4.0 Conclusions

The auto-radiotracer method gave excellent results in measuring the contaminants produced during this bearing test. Not only was it possible to determine the amount of the generated particles, but the origin of the material was revealed during the build-up of radioactivity.

TEST PLAN - BEARING TEST

1. Set up system in accordance with schematic (Figure 5-7).

2. Use preweighed collection filters downstream of bearing tester as follows:
   Three of each will be required:

   a) 80 x 700   (40 μ absolute)
   b) 325 x 2300   (10 μ absolute)

3. Fluid to be used - LN_2
   Source and quantity - LN_2 trailer, 1100 gallons.
   Pressure - 225 psig inlet
   Line size - 1/2 inch
   Total flow rate - 4 gpm
   Fluid inlet temperature - 160° R Max.

4. Install filter in pressure line. Use 325 x 2300 screen.

5. Use Bently or alternate speed pickup on end of shaft.

6. Open turbine seal drain to vent.

7. Establish $115^\pm$ psig GN_2 pressure in seal loading bellows cavity.

8. Start coolant flow at approximately 1 gpm to cool test to operating temperatures.

9. When tester is at operating temperature and coolant outlet is single phase, commence LN_2 flow through bearing cavity at approximately 0.8 gpm.

10. When LN_2 outlet from bearing cavity is single phase, adjust LN_2 flow to maintain 50 ± 10 psig in bearing cavity. Control using hand valve in outlet line.

11. Start GN_2 flow to impeller slowly and spin the shaft using a rubber tipped rod in a hand held, electric drill.
    (Note: Shaft rotates counter-clockwise, drill-reverse mechanism will be needed.)

12. Bring shaft slowly up to 20,000 rpm. Monitor LN_2 outlet temperature continuously to assure single phase flow through bearings.

13. Monitor activity of collection screens.

14. Replace screens after ninety minutes by diverting flow through parallel system.
    This will enable flow to be maintained.

15. Continue test until LN_2 is exhausted. The test duration will be known when optimum flow rates have been established.

16. If the test duration requires use of more than three screens of each size, the necessary additional screens should be cleaned and weighed early in the test program.

17. Care should be used in the transfer of the screens to petrie dishes in order that all contamination is removed on the screen.

18. Use a forty-minute count on all screens to give a good statistical value for the measured spectra.

19. To shut down tester, maintain LN_2 flow and cut off GN_2 flow to turbine. Following this, turn off main LN_2, seal loading pressure and LN_2 coolant flow in that order.

BEARING TEST SCHEMATIC
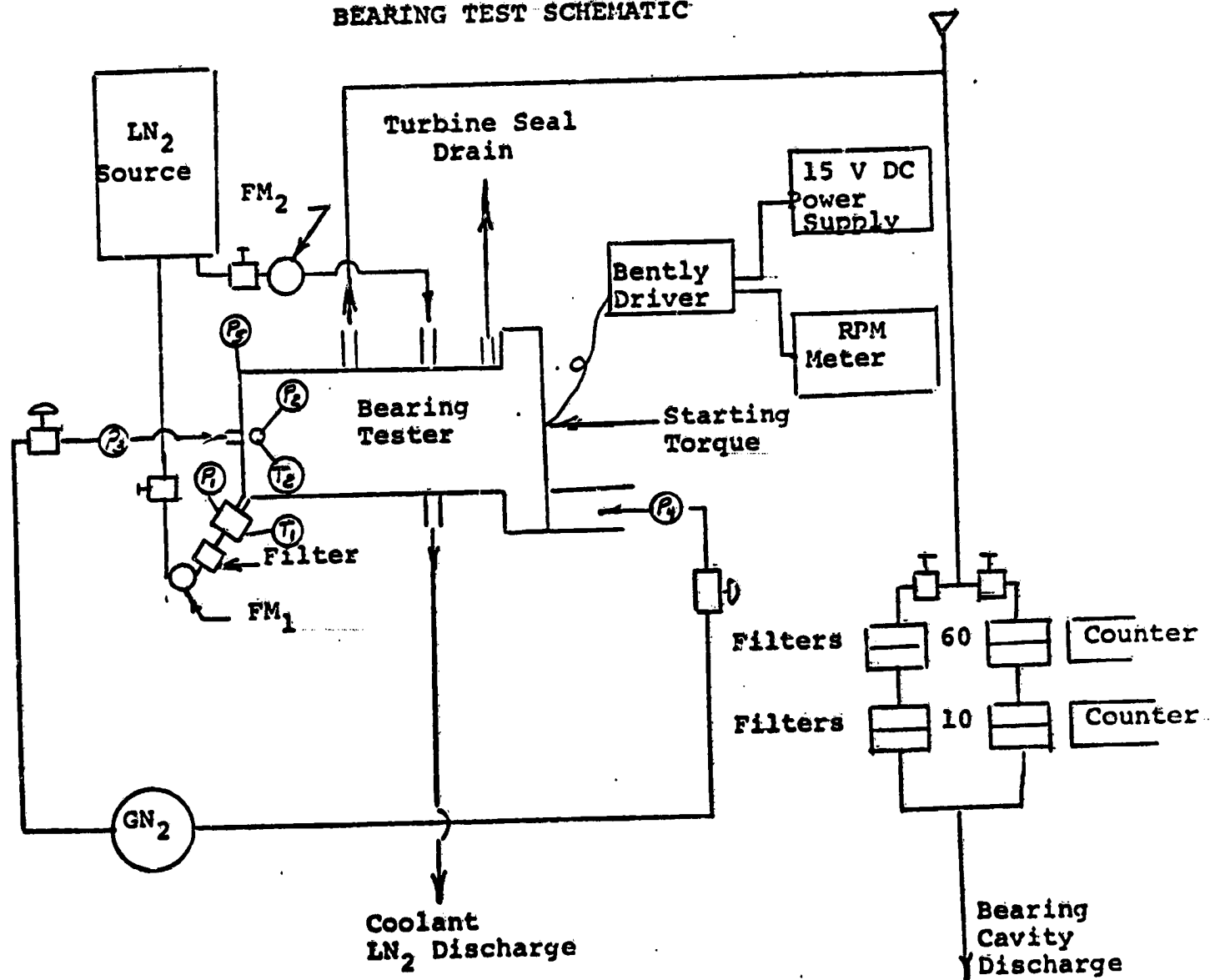
FM₁   Bearing Cavity Flow (1-2 gpm LN₂)

FM₂   Coolant Jacket Flow (2-5 gpm LN₂)

P₁   LN₂ Inlet Pressure (320 ± 10 psig)

P₂   LN₂ Outlet Pressure (40 ± 10 psig)

P₃   Seal Loading Bellows Pressure (115 ± 5 psig)

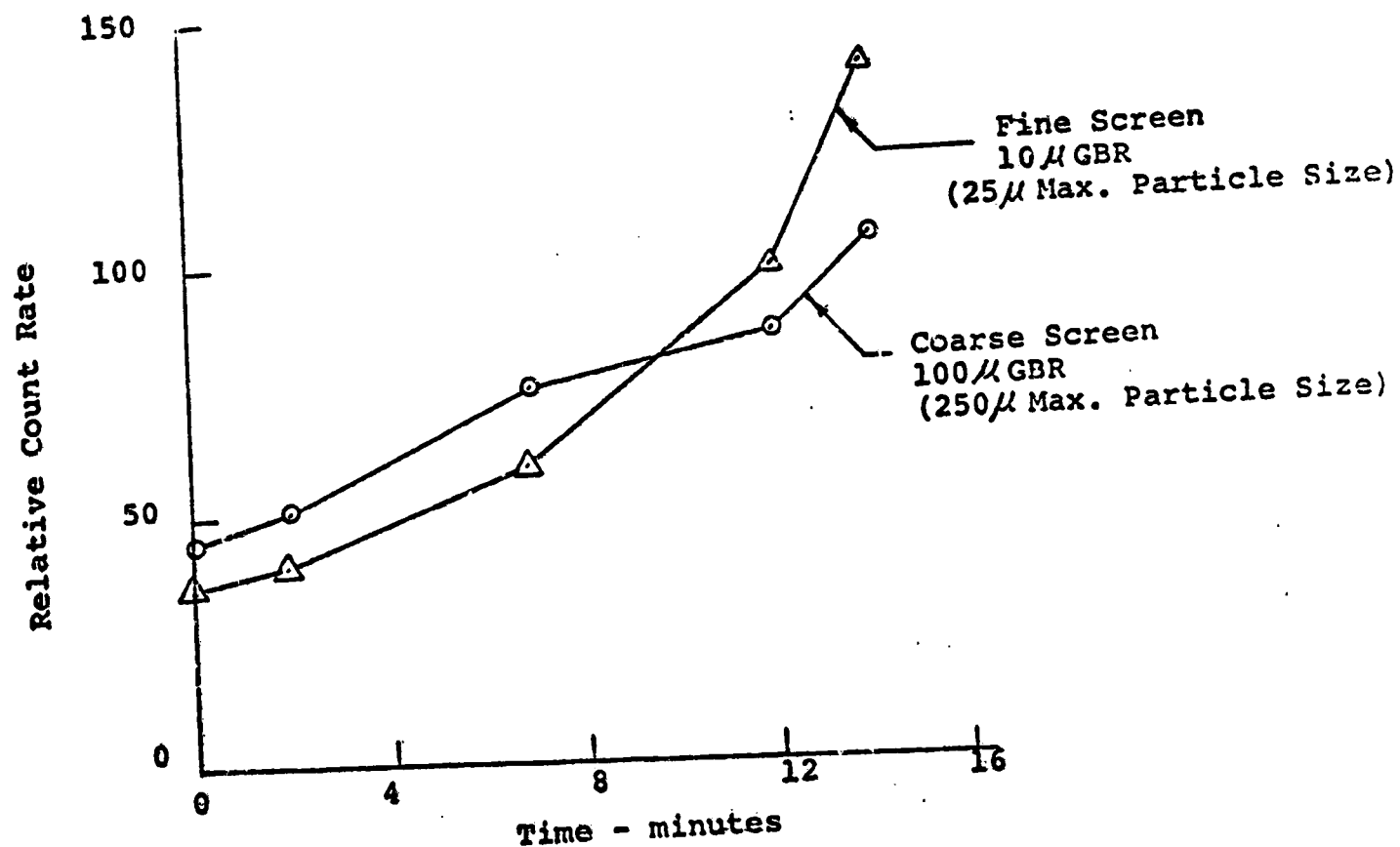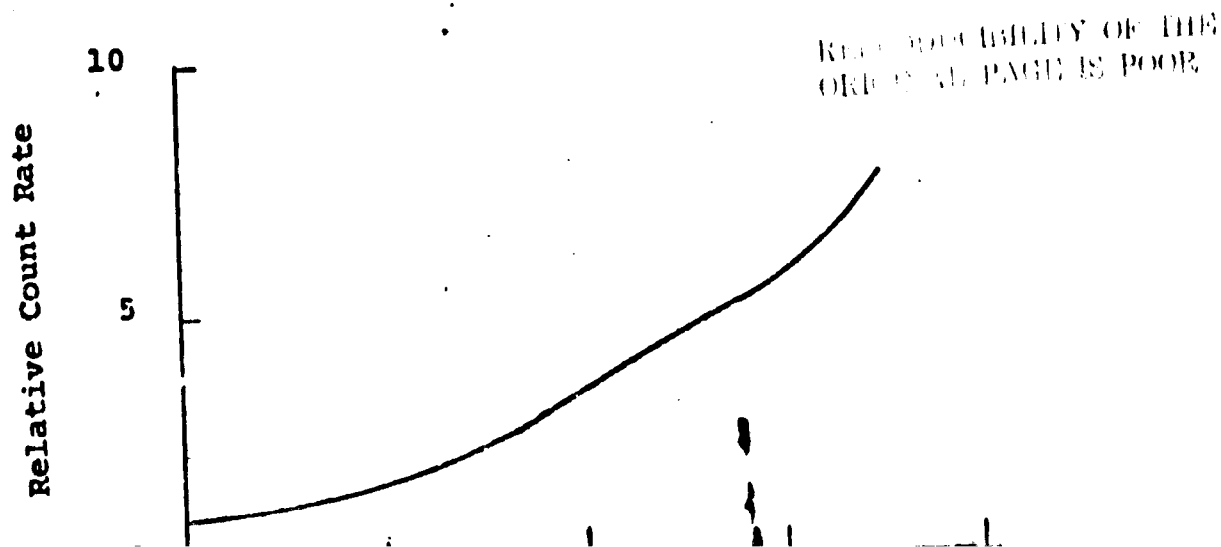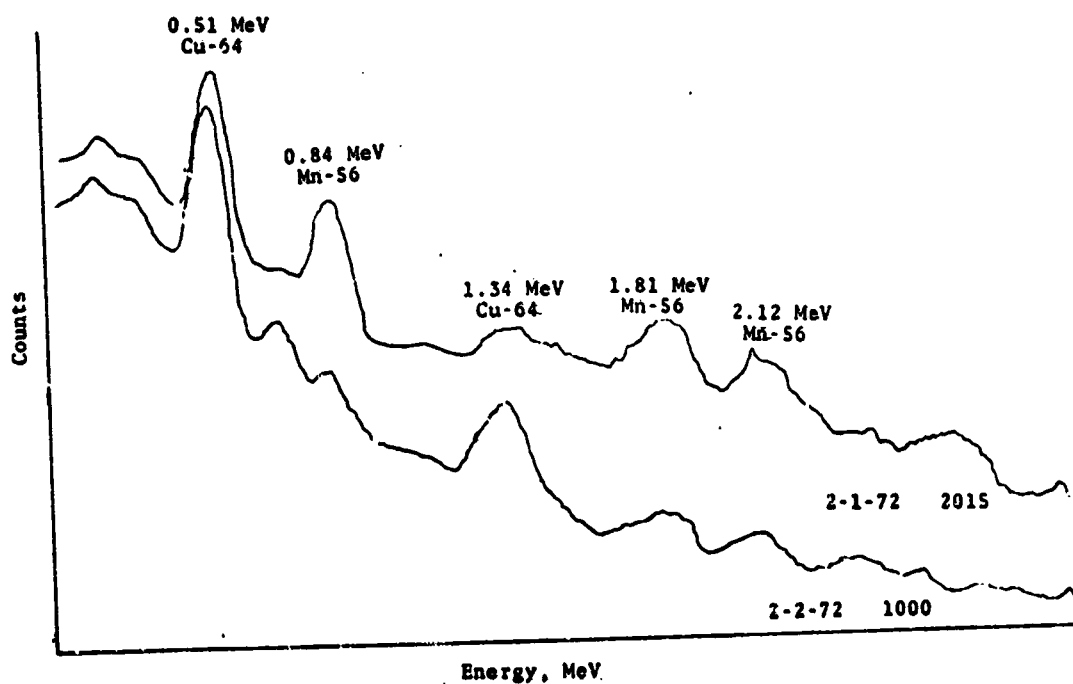FIGURE 5-2:   CONTAMINANT GENERATION – BEARING TESTS

ON-LINE METER INDICATIONS

Fine Screen
10 μ GBR
(25μ Max. Particle Size)

Coarse Screen
100μ GBR
(250μ Max. Particle Size)

Relative Count Rate

Time – minutes

FIGURE 5-3
ON-LINE STRIP-CHART RECORD

Relative Count Rate

FIGURE 5-4

GAMMA SPECTRA OF THE FINE SCREEN

(325 x 2300 TDDW - 10μ Absolute)

FIGURE 5-5

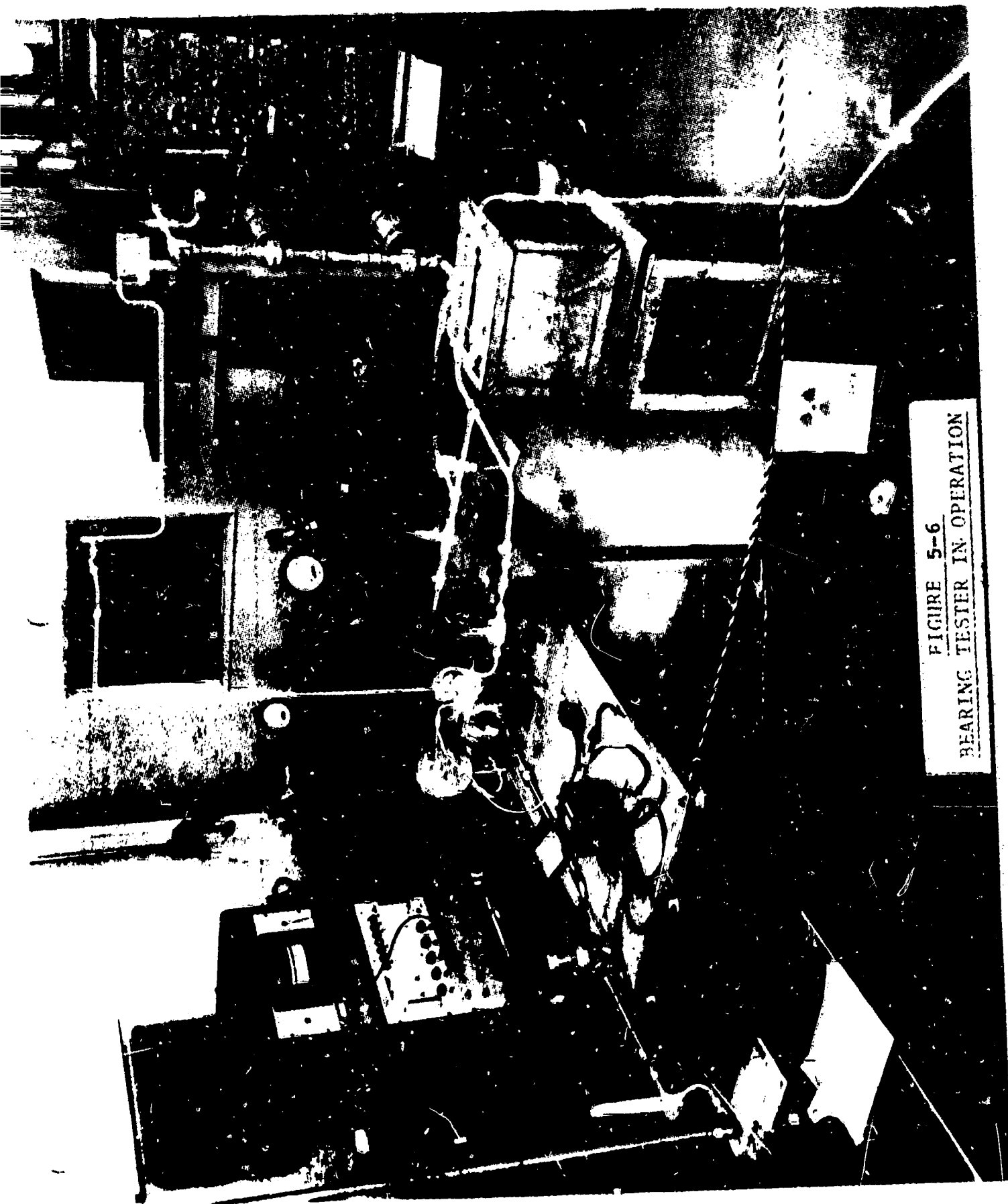**GAMMA SPECTRA OF THE COARSE SCREEN**

(80 x 700 TDDW - 40μ Absolute)
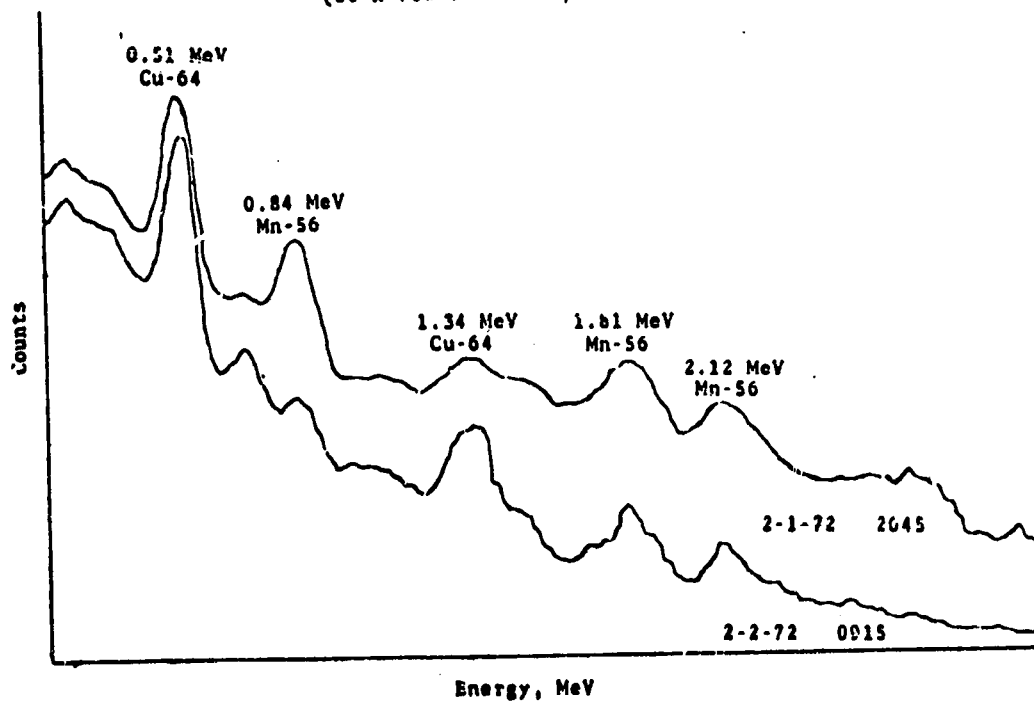
FIGURE 5-6
BEARING TESTER IN OPERATION

TEST NUMBER: 6

TEST ITEM: Various Plastic Materials

1.0 Scope

The purpose of this work was to determine the trace elemental constituents of various commonly used valve seat and bearing cage material samples. The analysis indicates which of the materials contain appropriate radio-isotopes, following irradiation, which may be used as radiotracers in short-term contamination generation studies. The materials analyzed are listed in Table 6-1.

2.0 Procedure

Thirteen material samples were irradiated in a neutron flux of 1.2×10^{12} n/cm^2-sec. for a period of 3 hours. Prior to irradiation, the samples were carefully cleaned, sealed in plastic bags in a clean room, and re-sealed in cleaned plastic containers. Prior to counting the irradiated samples, the material samples were removed from the plastic bags and original plastic containers and then transferred to unirradiated containers. This procedure insured that the analysis revealed only those trace constituents contained in the material samples themselves and not in the sample containers or surface contamination.

Following removal from the reactor, the samples were counted using a sodium-iodide (NaI) scintillation crystal and photomultiplier tube and a 400 channel pulse height analyzer. Counts were taken at various times following irradiation to determine both the characteristic gamma energies and the half-lives of the various trace constituents of each of the samples.

3.0 Results

Table 6-2 summarizes the results of the activation analysis. The activity values shown in the table are activity levels of the specific radionuclides contained in one gram of the material sample upon removal from the reactor.

The Nylatron GS, Kel F 81, Rulon-J, Armalon, Fluorocreen E-600 and reprocessed teflon samples may have also contained Mn-56. However, due to the relatively high activity of these samples upon removal from the reactor, it was not possible to count the samples until the short-lived isotopes had decayed. Manganese-56 and perhaps silicon-31 are suspected to be the isotopes responsible for the short-lived activity of these samples.

Gamma spectra for the thirteen materials are shown in Figures 6-1 through 6-13. For the virgin Teflon and Kel-F 81 samples it is indicated in Figures 6-1 and 6-6 respectively that what appears to be a single peak is actually due to the presence of two different

isotopes. Each of the isotopes has a distinct gamma energy level, although the energy difference is slight. For the virgin Teflon (Figure 6-1) the two isotopes are Bi-31 (1.27 MeV) and Na-24 (1.37 MeV). For the Kel-F 81 (Figure 6-6) the isotopes are Cu-64 (0.51 MeV) and W-187 (0.48 MeV). For both materials one of the paired isotopes has a significantly shorter half-life than the other; as the shorter lived isotope decays the contribution from the longer lived isotope will predominate. It is the difference in half-lives which facilitates the identification of both isotopes contributing to what appears to be a single peak.

4.0 Conclusions

The cleaning procedures that were followed, prior to irradiation, insured that the trace constituents found in the analysis were actually constituents of the material samples and were not due to surface contamination. The results indicate that Mn-56, Cu-64 and Na-24 are common to most of the samples analyzed. It has yet to be determined whether these are characteristic constituents of the materials or whether they are picked up in processing the material and component fabrication. Regardless of the source, however, both Cu-64 and Na-24 have adequate half-lives (12.8 hr and 15 hr respectively) to allow short-term wear studies to be conducted. This is, of course, providing that the specific activities of the irradiated component materials are known immediately prior to conducting the test and that the components can be irradiated, assembled, and tested within a period of approximately two days.

The Kel F 81 sample was found to contain W-187 (half-life = 24 hr); the Delrin sample was found to contain Au-198 (half-life = 64.8 hr); and the N latron CS sample was found to contain Mo-99 (half-life = 66 hr). While none of these radioisotopes has a sufficient-ly long half-life to be used as a radiotracer in a long-term generation study, all have half-lives significantly longer than those of both Cu-64 and Na-24. This will allow the performance of contaminant generation tests over periods of greater than the two-day time limitation for materials containing only the shorter-lived isotopes. Using W-187 as a radiotracer in a contamination generation study, the corresponding time restriction would be 3 days; and for Au-198 and Mo-99 it would be 8 days.

Specific activities somewhat higher than those achieved in the 3-hour irradiation would be required to obtain significant data in a longer term contaminant generation study in-volving the materials analyzed. From the results presented, the period of irradiation required to achieve the desired activity level may be extrapolated. For a typical long term contaminant generation test, the initial component activity should be on the order of 100 Ci.

Utilizing the longer half-lives of W-187 and particularly Au-198 and Mc-99, the respective materials may be irradiated to the desired specific activity level of o a of these isotopes. After 3 days, the shorter lived isotopes will have essentially decayed away and only the longer-lived isotope will remain at appreciable activity levels.

The Rulon-J sample contained approximately 10 Ci/gm of Cd-115 (half-life 53.5 hr), and the Fluorogreen E-600 sample contained approximately 0.5 Ci/gm of Cr-51 (half-life 27.8 days). Both of these isotopes are promising for contamination tests. The Cr-51 specific activity in the Fluorogreen E-600 could easily be increased by extending the irradiation period. Six hours irradiation would yield approximately 1 Ci/gm of Cr-51.

The Cr-51 which develops by irradiation of Fluorogreen E-600 makes this material especially promising for long term tests. The material can be irradiated to the desired specific activity level of the Cr-51 and allowed to stand before use so that essentially all of the shorter lived isotopes will decay away, leaving only the Cr-51 at very nearly its initial specific activity level.

TABLE 6-1

LIST OF SAMPLE MATERIALS

| Sample Material	Mass (gm)
1. Virgin teflon	6.60
2. Kynar	1.41
3. Vespel	5.55
4. Teflon FEP	6.31
5. Nylatron GS	6.93
6. Kel F 81	5.80
7. Reprocessed teflon	27.75
8. Nylon LP 410 6/6	7.43
9. Delrin	3.98
10. Rulon-J	0.88
11. Armalon	2.85
12. Virgin Teflon 25% glass-filled	16.68
13. Fluorogreen E-600	15.94

TABLE 6-2

SUMMARY OF ACTIVATION ANALYSIS RESULTS

Sample Identification	Isotope	Composition Half-life (hr)	Activity (Ci/gm)
1. Virgin Teflon	Mn-56	2.58	0.3
	Si-31	2.62	0.07
	Cu-64	12.8	0.5
	Na-24	15.0	0.02
	Total		0.09
2. Kynar	Mn-56	2.58	0.1
	Cu-64	12.8	0.1
	Na-24	15.0	0.03
	Total		0.23
3. Vespel	Mn-56	2.58	5.9
	Cu-64	12.8	2.1
	Na-24	15.0	2.1
	Total		10.1
4. Teflon FEP	Mn-56	2.58	1.5
	Cu-64	12.8	1.5
	Na-24	15.0	0.7
	Total		3.7
5. Nylatron GS	Cu-64 —	12.8	1.4
	Na-24	15.0	0.25
	Mo-99	66.0	1.6
	Total		3.2
6. Kel F 81	Cu-64	12.8	4.0
	Na-24	15.0	3.7
	W-187	24.0	4.3
	Total		12.0
7. Reprocessed Teflon	Cu-64	12.8	4.3
	Na-24	15.0	3.4
	Total		7.7
8. Nylon LP 410 6/6	Mn-56	2.58	2.2
	Cu-64	12.8	0.6
	Na-24	15.0	0.3
	Total		3.1
9. Delrin	Mn-56	2.58	0.09
	Cu-64	12.8	0.06
	Na-24	15.0	0.03
	Au-198	64.8	0.005
	Total		0.18
10. Rulon-J	Na-24	15.0	13.4
	Cd-115	53.5	10.1
	Sc-46	83.8 day	0.01
	Sc-75	120.0 day	0.35
	Total		23.9
11. Armalon	Cu-64	12.8	155.0
	Na-24	15.0	126.0
	Sc-46	83.8 day	0.1
	Total		281.1
12. 25% Glass Filled Teflon	Cu-64	12.8	48.8
	Na-24	15.0	51.4
	Sc-46	83.8 day	0.02
	Total		100.2
13. Fluorogreen E-600	Cu-64	12.8	60.8
	Na-24	15.0	66.5
	Cr-51	27.8 day	0.5
	Sc-46	83.8 day	0.008
	Total		127.8

FIGURE 6-1

GAMMA ENERGY SPECTRUM - IRRADIATED VIRGIN TEFLON
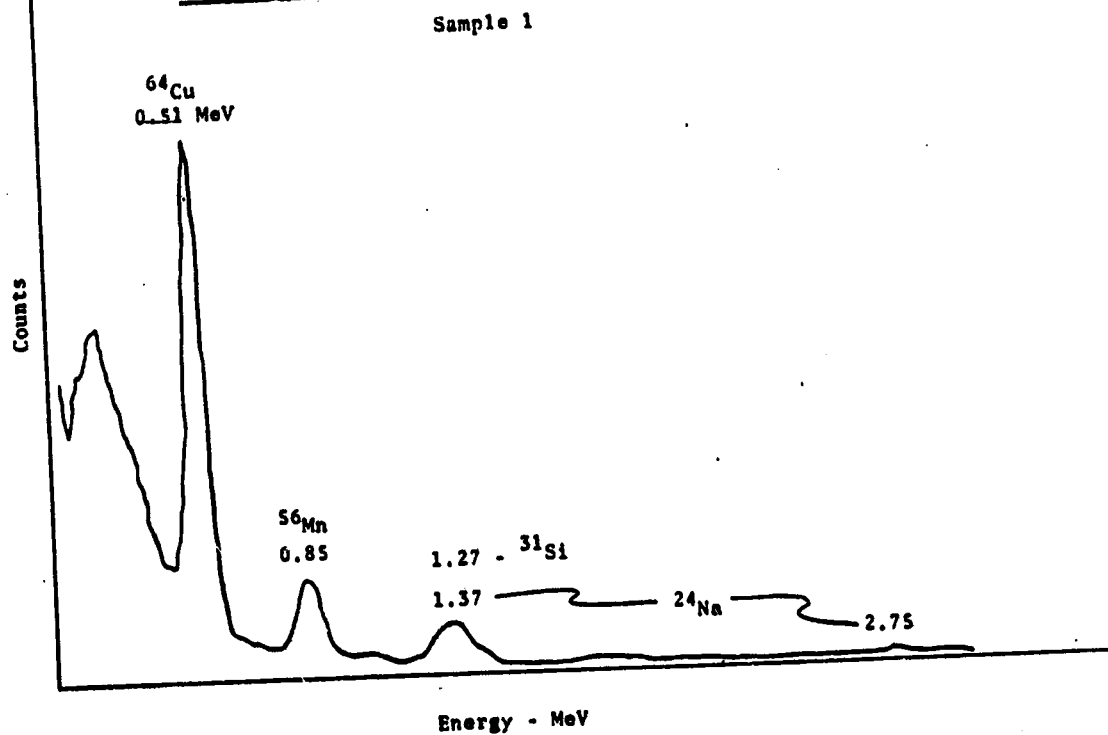
Sample 1

FIGURE 6-2

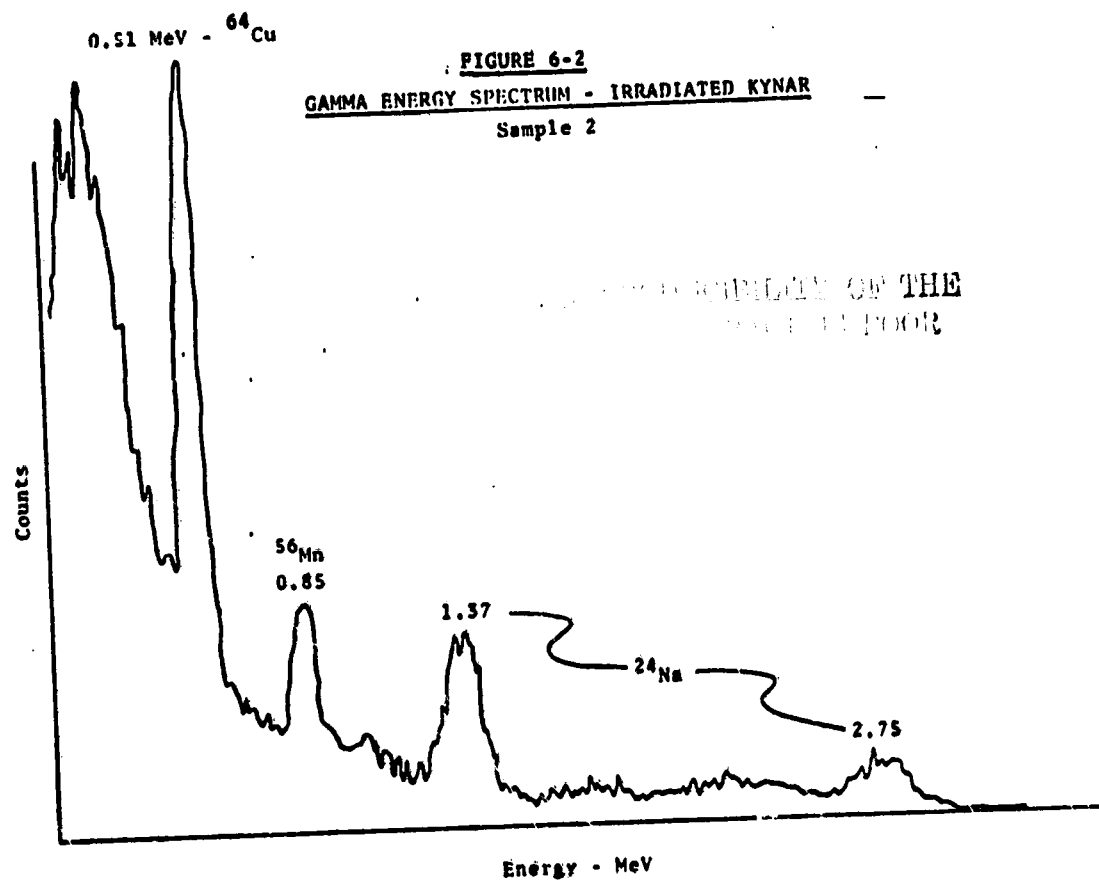## GAMMA ENERGY SPECTRUM - IRRADIATED KYNAR

Sample 2

91

FIGURE 6-3

GAMMA ENERGY SPECTRUM - IRRADIATED VESPEL
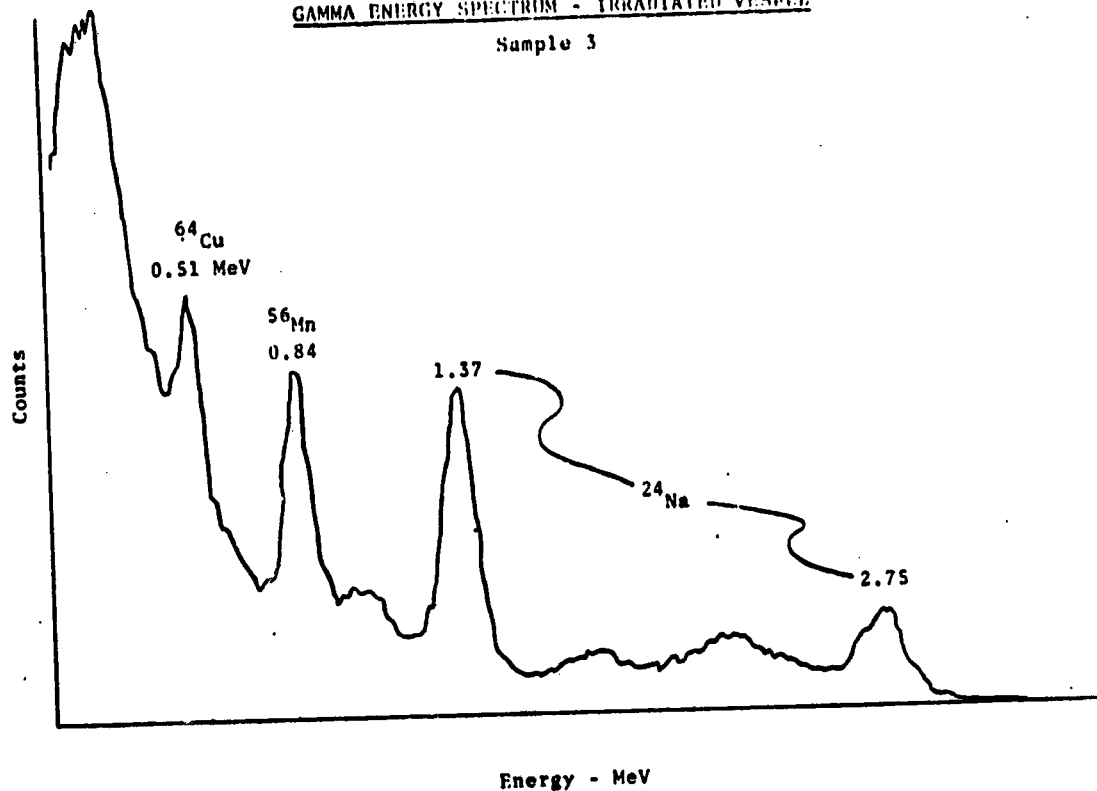
Sample 3

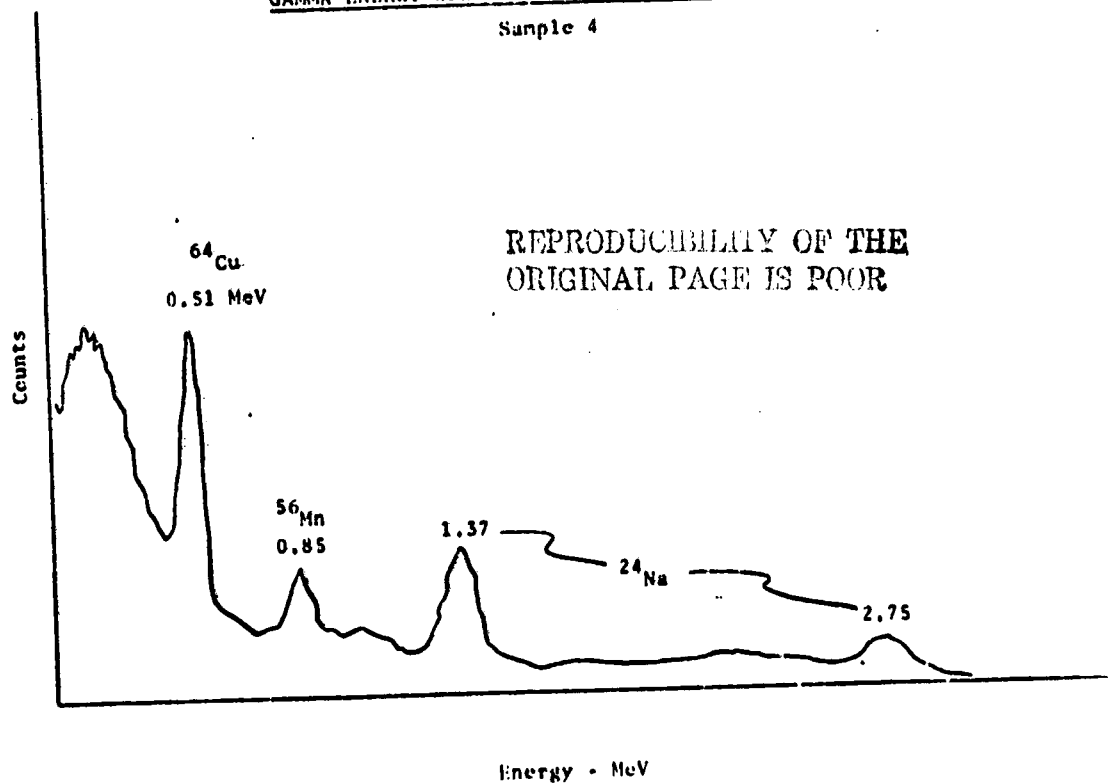Energy - MeV

FIGURE 6-4

GAMMA ENERGY SPECTRUM - IRRADIATED TELF IN FEP
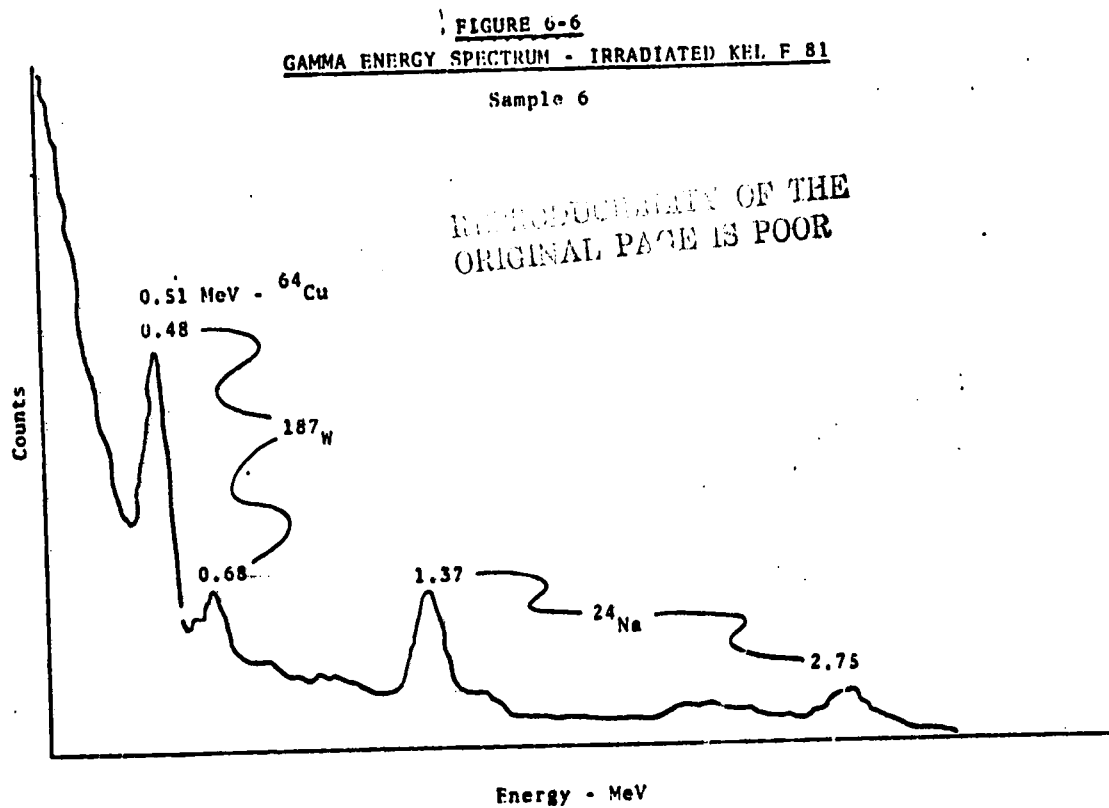
Sample 4

REPRODUCIBILITY OF THE
ORIGINAL PAGE IS POOR

Energy - MeV

FIGURE 6-5

GAMMA ENERGY SPECTRUM - IRRADIATED NYLATRON GS

Sample 5

FIGURE 6-6

GAMMA ENERGY SPECTRUM - IRRADIATED KEL F 81
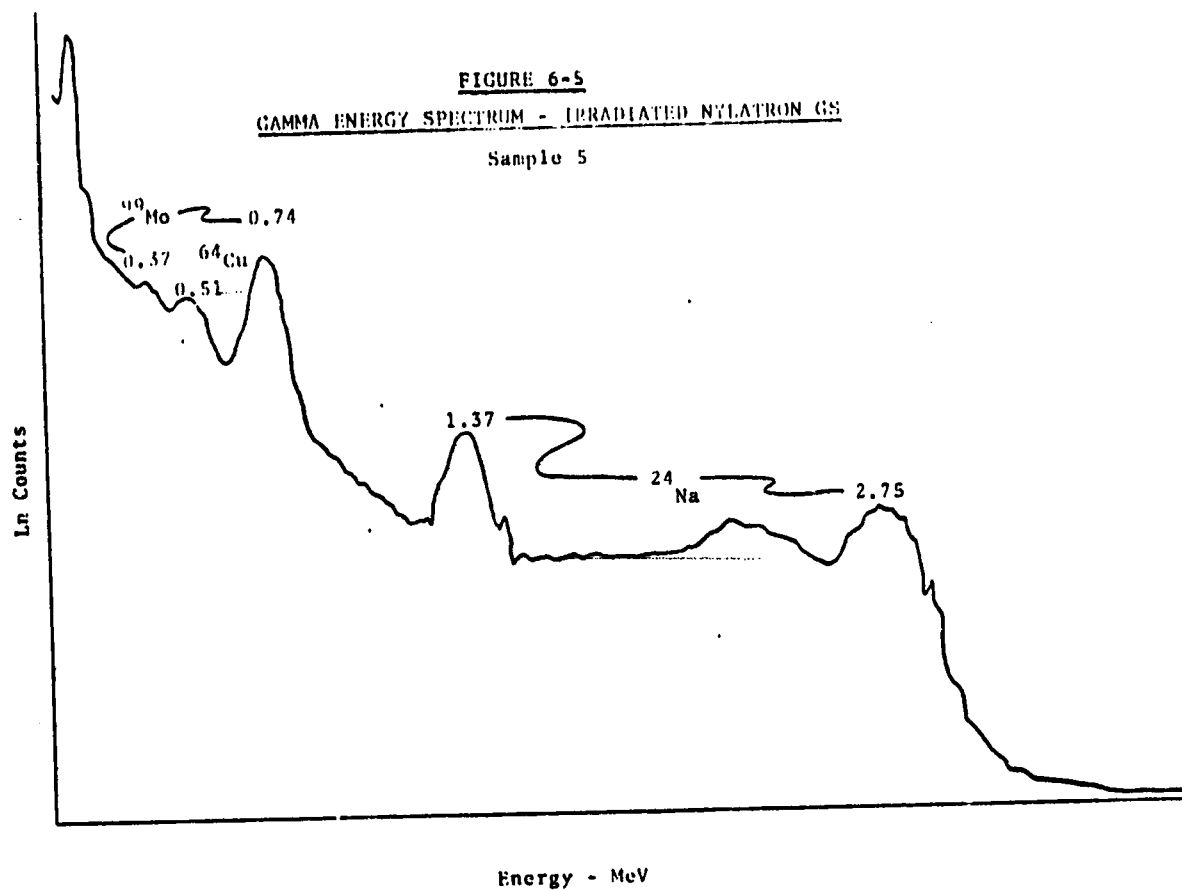
Sample 6

FIGURE 6-7

GAMMA ENERGY SPECTRUM - IRRADIATED REPROCESSED TEFLON

Sample 7

Energy - MeV
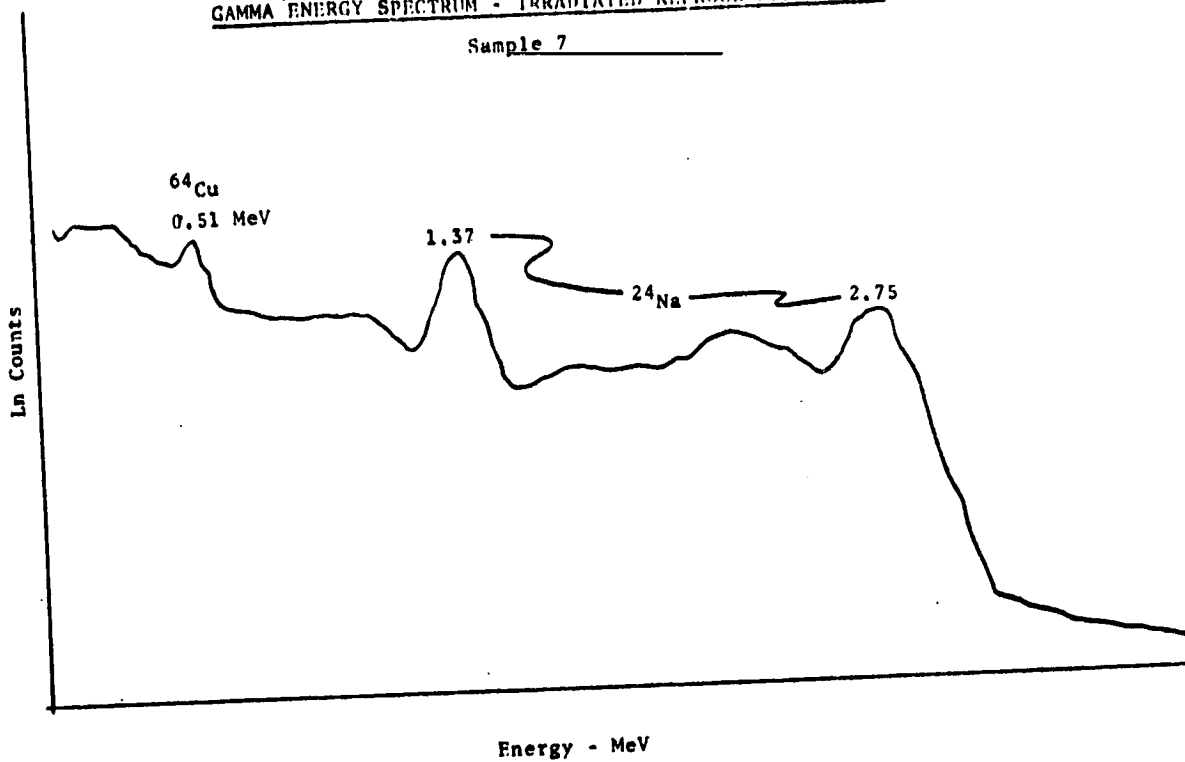
FIGURE 6-8

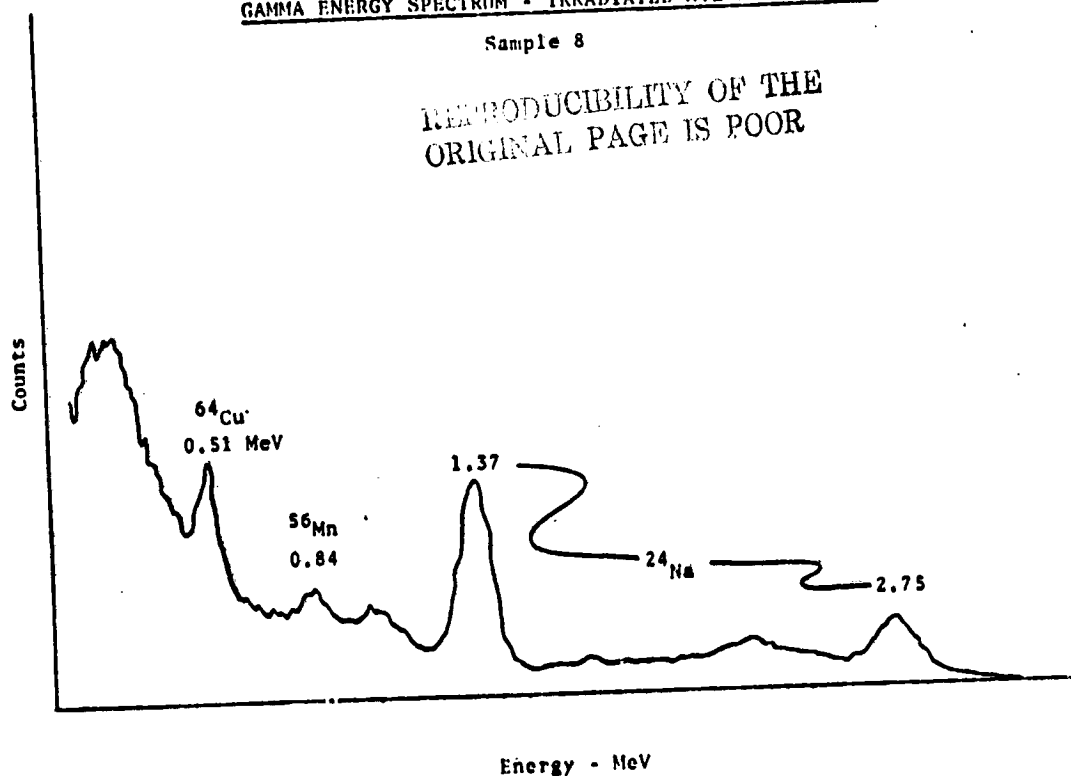GAMMA ENERGY SPECTRUM - IRRADIATED NYLON LP410 6/6

Sample 8

Energy - MeV

FIGURE 6-9

GAMMA ENERGY SPECTRUM - IRRADIATED DELRIN

Sample 9

Energy - MeV

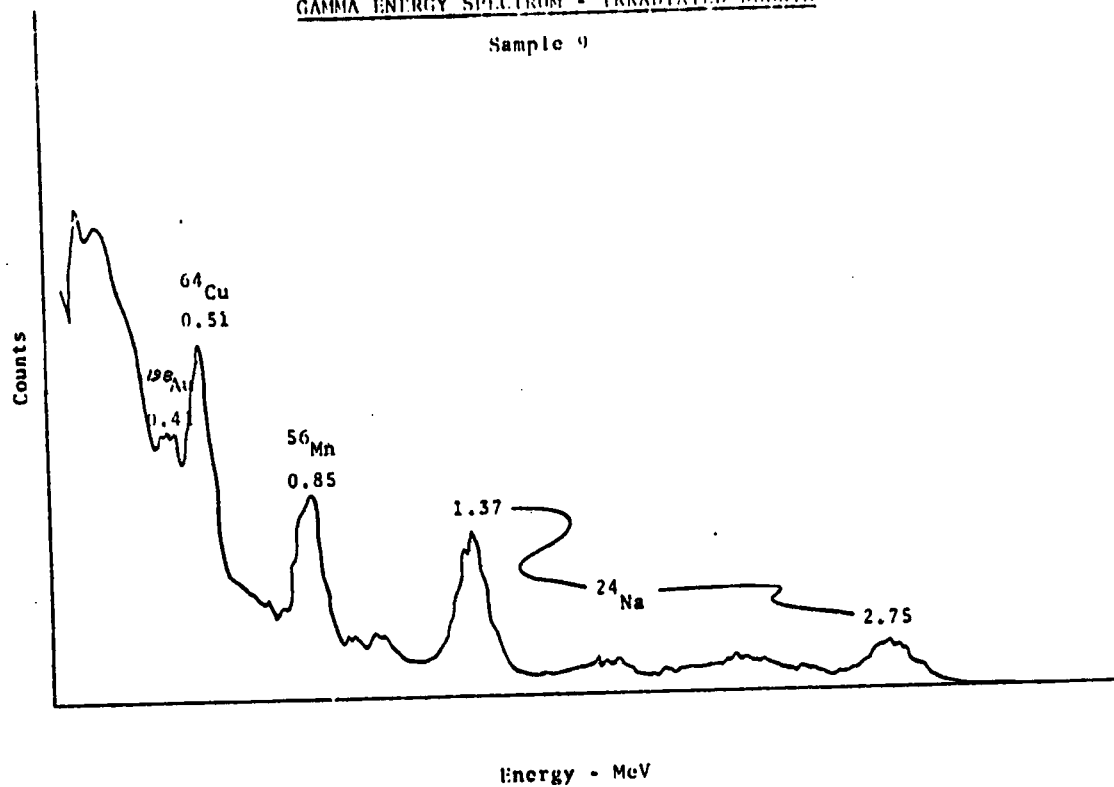FIGURE 6-10

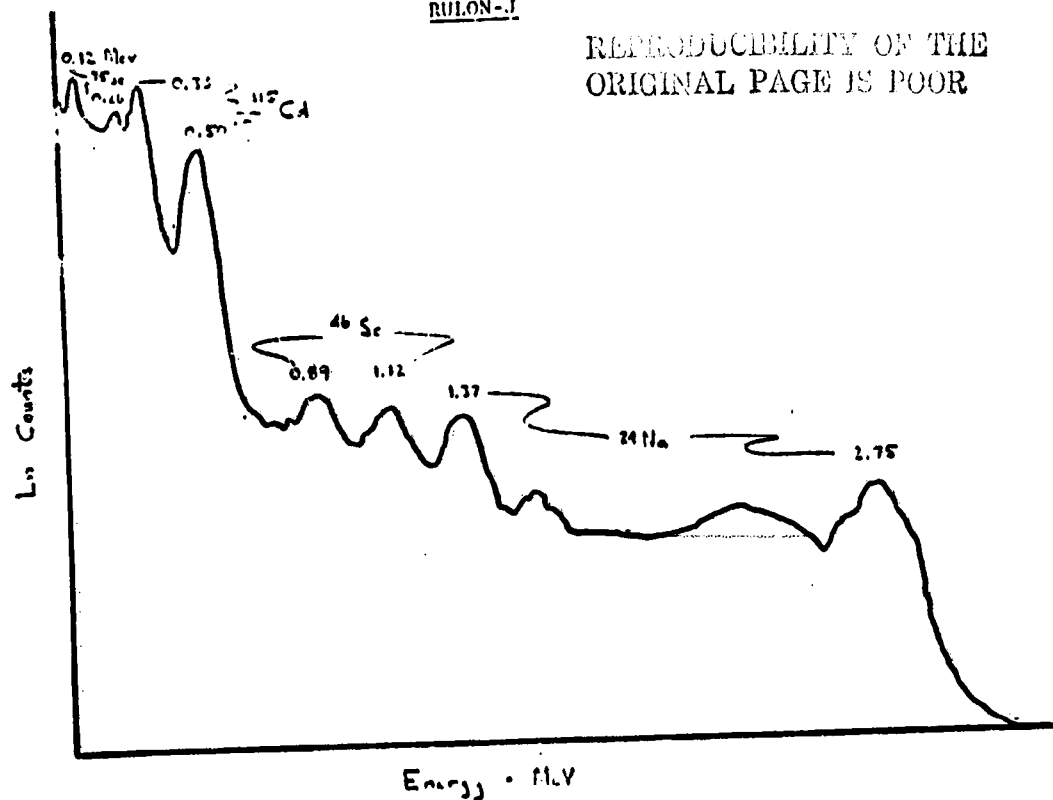RULON-J

REPRODUCIBILITY OF THE
ORIGINAL PAGE IS POOR
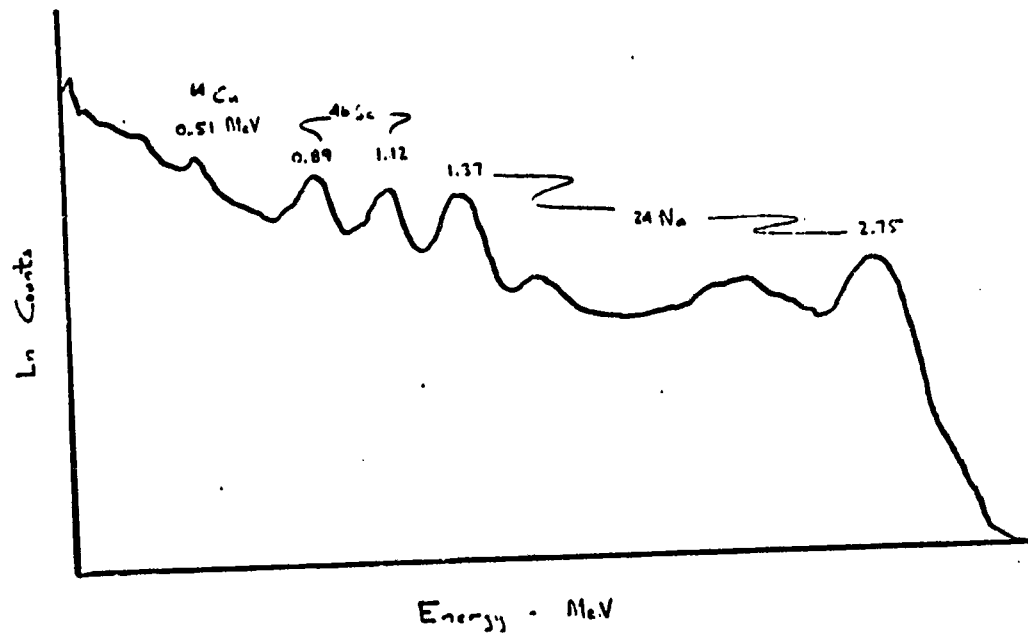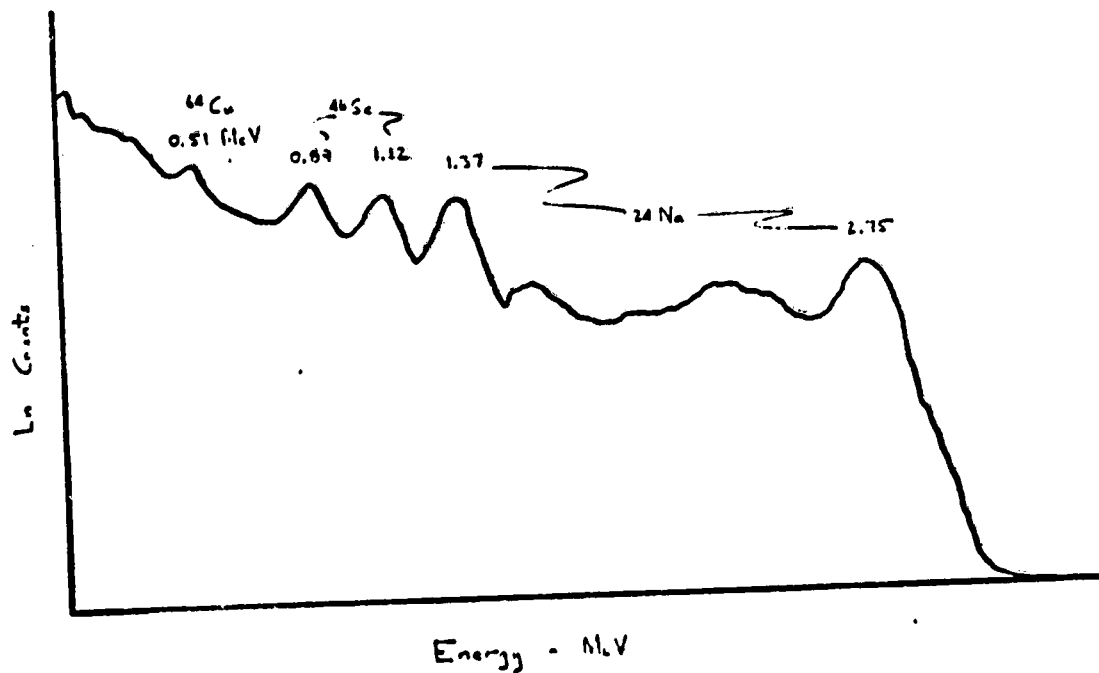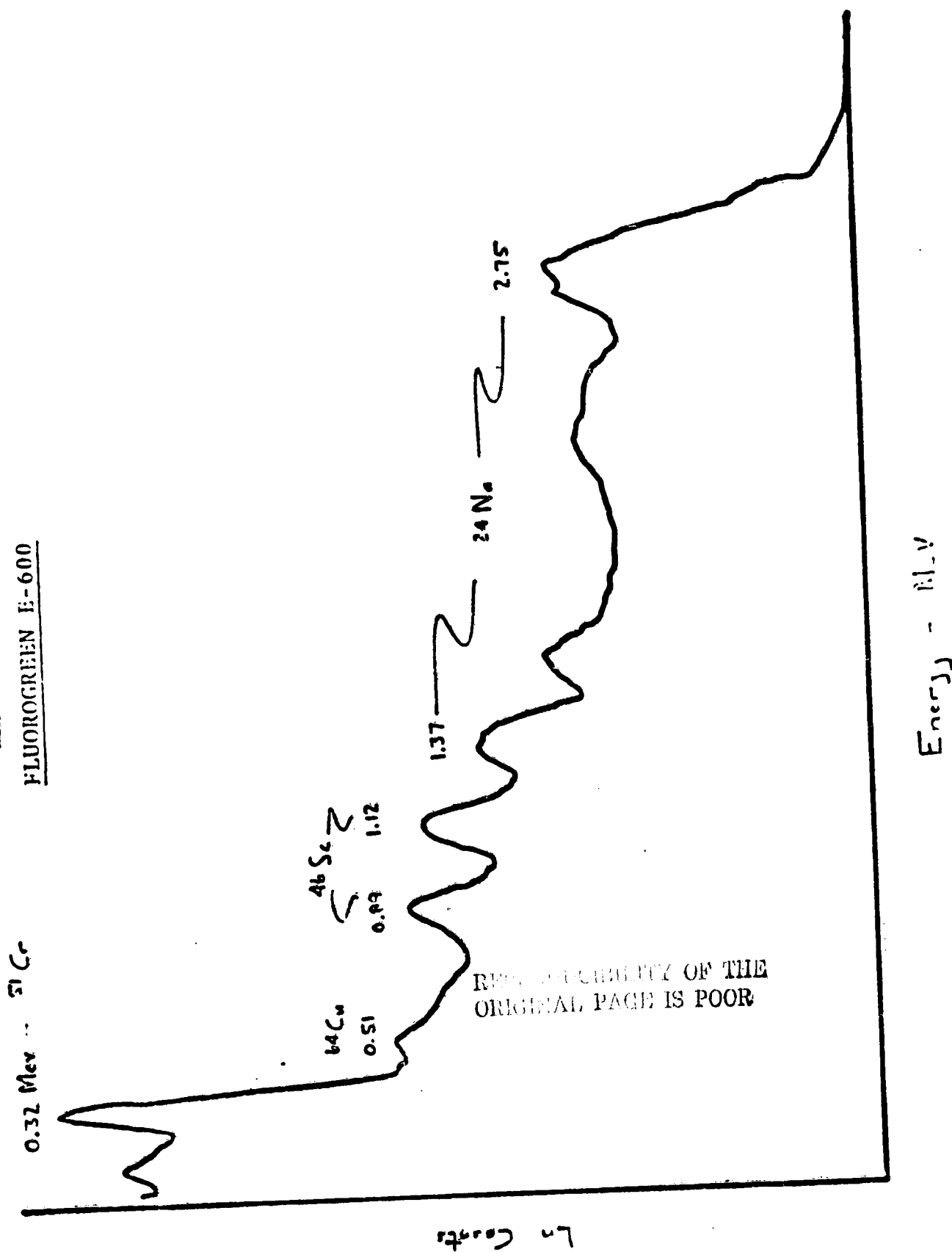
Energy - MeV

FIGURE 6-11
ARMALON

FIGURE 6-12
25% GLASS-FILLED VIRGIN TEFLON

FIGURE 6-13

FLUOROGREEN E-600

TEST NUMBER: 7

TEST ITEM: 3/4" Teflon Seal Ball Valve

1.0 Scope

The purpose of this test was to determine the contamination generation characteristics of a 3/4" ball valve equipped with teflon seals. Tests of particle generation were conducted in both gas and liquid systems. As relatively large amounts of generated material were anticipated, the gravimetric method was used. Figure 7-1 shows a cross section of the ball valve.

2.0 Procedure

2.1 Gas Test

2.1.1 Preparation of Test Item

Prior to beginning the test, the valve, consisting of a 303 stainless steel ball, stem and body and Teflon primary and stem seals was disassembled, ultrasonically cleaned and reassembled.

2.1.2 Test Description

The valve was installed in a test system consisting of a nitrogen source, a pressure regulator, a 10 micron filter immediately upstream of the test valve, the test valve and a 10 micron (325 x 2300) collection screen immediately downstream of the test valve. Four (4) pre-cleaned, dried and weighed collection screens were provided, as it was planned to perform the test in four (4) increments of 100, 100, 500 and 1000 cycles. A pneumaticlly operated actuator and counter was installed to provide 20 on-off cycles of the test valve per minute. The regulator was set to deliver nitrogen to the test valve at 100 pisg.

The first test consisted of 100 on-off cycles. At the conclusion of this test the collection screen was carefully removed and replaced with a clean screen. The collection screen was dried and weighed to determine the amount of contaminant collected in the first 100 cycles. This procedure was repeated three times at increments of 100, 500, and 1000 cycles for a total of 1700 cycles.

2.1.3 Results

Table 7-1 shows the weight of contaminant collected in each test increment. Figure 7-2 shows the test data presented in graph form. It can be seen that after a sharp rise in generation of material, the rate of generation became relatively constant as shown by the straight section of the graph. A large amount of material was released throughout the test, totalling 28 milligrams after 1700 cycles. After weighing the screens,

they were examined under a microscope at 40 and 100 power. The generated material consisted entirely of shreds and particles of Teflon ranging in size form 25 microns to 2000 microns. The largest particle measured 2080 x 1240 microns.

2.2 Liquid Test

2.2.1 Preparation of Test Item

New main seals and stem seal were provided and the valve was prepared for test as in 2.1.1.

2.2.2 Test Description

The valve was installed in a liquid test system using water as the test fluid. The system was similar to that used for the gas test except that the nitrogen source was replaced by a reservoir and pump. A flow control valve set to deliver 2 gpm of water replaced the pressure regulator.

The test was conducted in five (5) increments of 100, 100, 500, 1000, and 1000 cycles for a total of 2700 cycles. The collection screen was removed and weighed after each increment.

2.2.3 Results

Table 7-1 shows the weight of contaminant collected in each test increment. Figure 7-2 shows the plotted test data. The generation rate remained relatively constant until 1700 cycles as shown by the straight section of the plot and then decreased during the last test increment. The total amount of material generated in 1700 cycles was approximately twice that collected in the gas test.

Microscopic examination showed the generated material to be Teflon shreds and particles with sizes varying from 25 microns to 2000 microns as was observed in the gas test.

3.0 Conclusions

When the valve was disassembled and examined after both tests, it was apparent that the source of the Teflon material was the stem and seal and not the primary ball seals. The stem seal is compressed by a clamp plate forcing the Teflon to cold-flow and form a seal with the valve stem. As the Teflon cold-flows, it also extrudes through the clearance space between the stem and the body. As the valve is cycled the thin extrusion breaks off and falls into the cavity between the flat sides of the ball and the body. Subsequent rotation of the ball through 90 degrees "sweeps" the shavings into the flow stream and where they are collected on the downstream filter.

TABLE 7-1

CONTAMINANT GENERATION TEST - 3/4" BALL VALVE

Gas Test (Nitrogen)

Run Number	Number of Cycles		Weight of Contaminant mg.	
	Per Run	Cumulative	Per Run	Cumulative
1	100	100	6.6	6.6
2	100	200	4.1	10.7
3	500	700	5.5	16.2
4	1000	1700	11.8	28.0

Liquid Test (Water)

Run Number	Number of Cycles		Weight of Contaminant mg.	
	Per Run	Cumulative	Per Run	Cumulative
1	100	100	2.0	2.0
2	100	200	2.7	4.7
3	500	700	16.1	20.8
4	1000	1700	29.0	49.8
5	1000	2700	16.2	65.0

303 CRES Ball
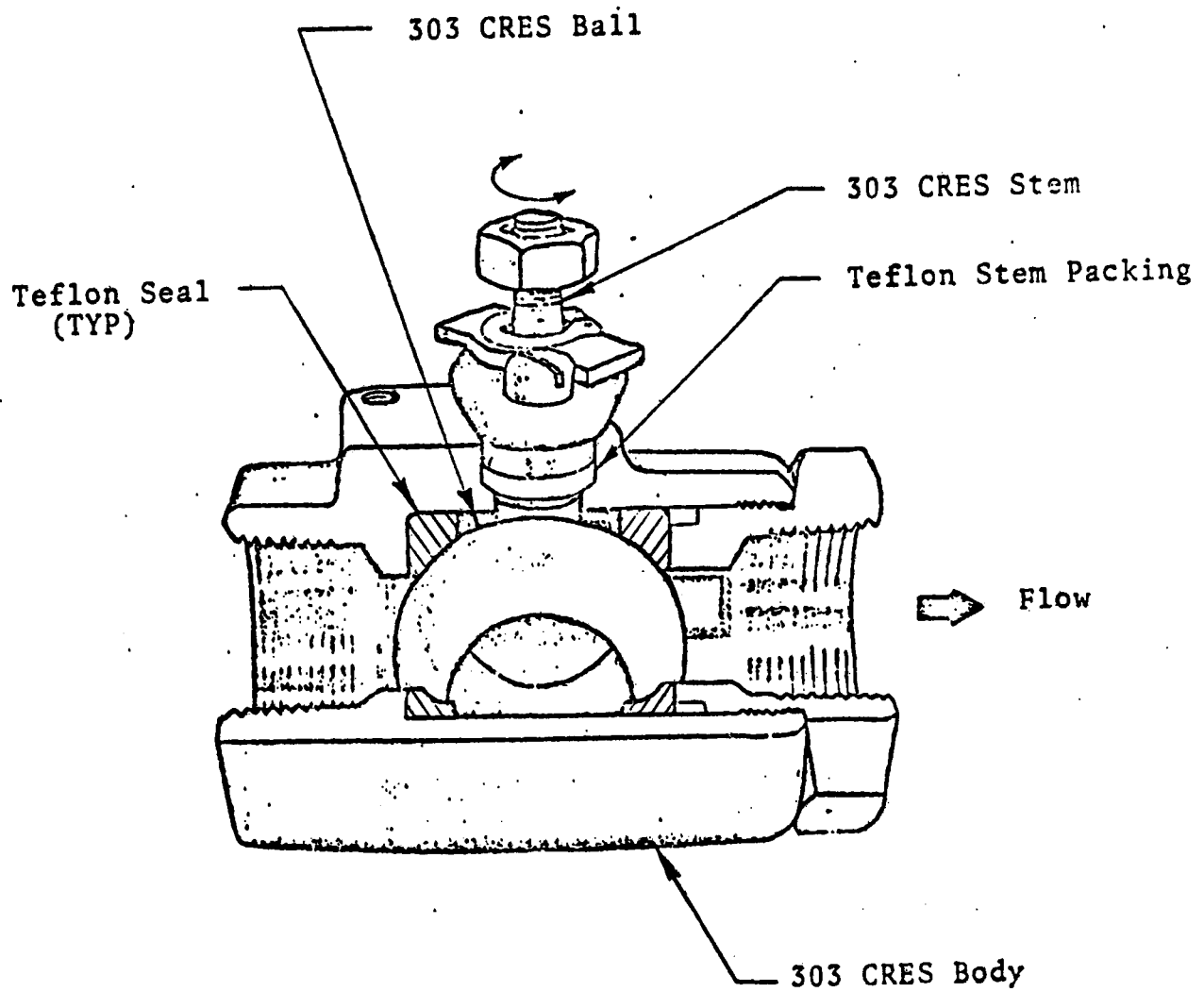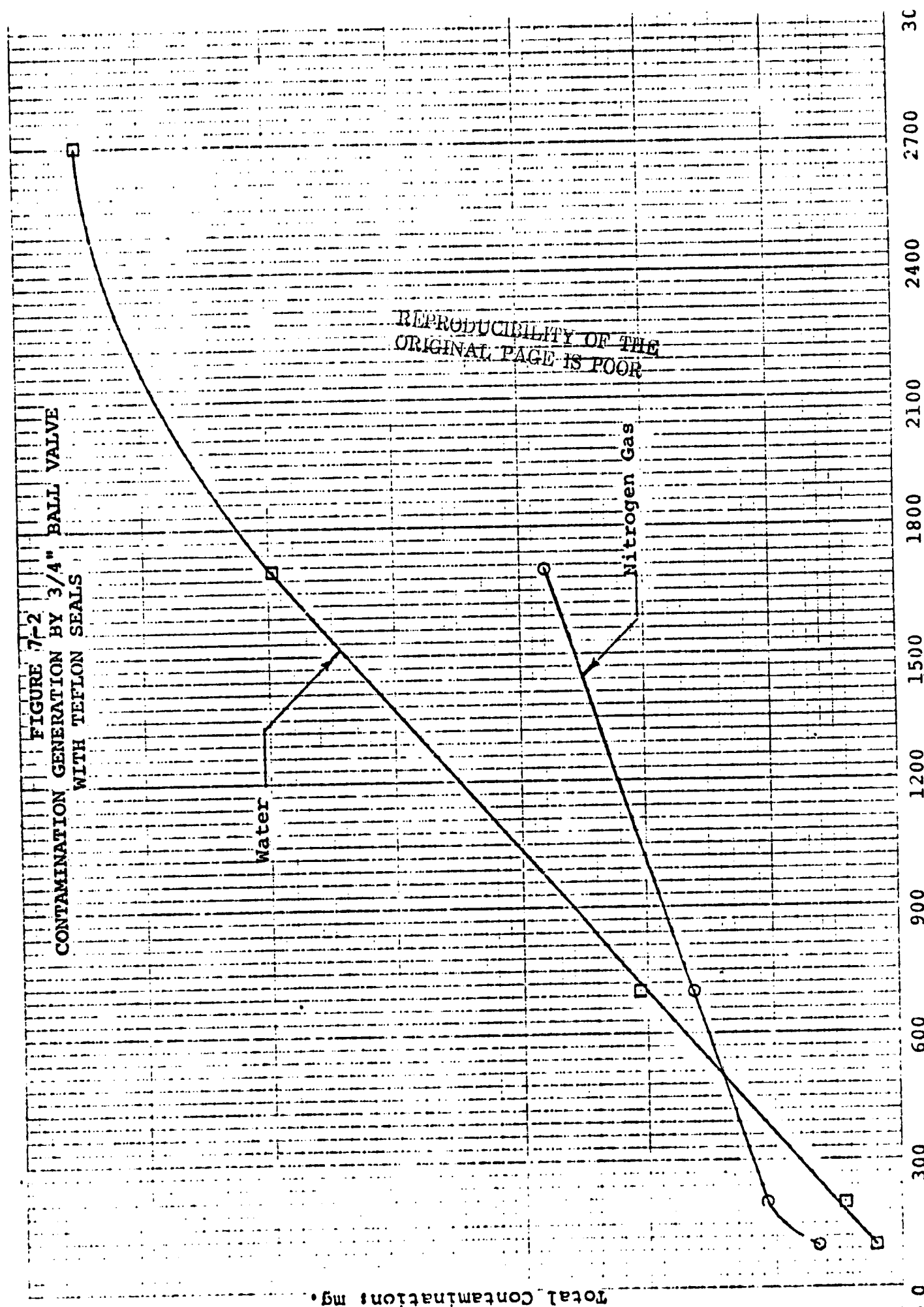
303 CRES Stem

Teflon Seal
(TYP)

Teflon Stem Packing

Flow

303 CRES Body

FIGURE 7-1

3/4 INCH BALL VALVE

FIGURE 7-2

CONTAMINATION GENERATION BY 3/4" BALL VALVE WITH TEFLON SEALS

SECTION B

COMPONENT SENSITIVITY TESTS

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1.0 INTRODUCTION

The selection of the optimum type and grade of filter medium to be used in a fluid system is dependent on the characteristics of the operating components in the system. Those components which are very sensitive to contamination require very clean operating fluids and therefore a finer degree of filtration than those relatively insensitive to particulate contamination. The optimum choice of filter medium is that which will provide only the degree of protection required, as filter media which provide finer filtration than required will, of necessity, be larger than optimum due to the direct relationship between filter rating and contaminant tolerance. The finer the filtration rating, the more screen area, i.e. filter size, is required to ingest a given amount of contaminant.

In order to determine contaminant sensitivity of any given component, the unit can be operated in a contaminated flowing system sequentially protecting it with upstream filters of four standard filtration ratings. Starting with the finest filtration rating (10/25) for the first cycle, additional life cycle tests are conducted with the 20/50, 40/100 and 100/250 filter media until failure occurs. The largest micron-rating medium with which the component can perform its design life cycle without failure or performance degradation below allowable limits represents the optimum choice of filter medium to be used in the protective filter.

2.0 TEST METHOD - GENERALIZED DESCRIPTION

The four grades of "standard" filter media recommended in Volume I of this report are as follows.

Medium (Nominal Grade)	Glass Bead Rating (Microns)	Maximum Particle Size Rating (Microns)
325 x 2300 TDDW	10	25
2 x 120 x 650 PDSW	20	50
80 x 400 PDSW	40	100
30 x 160 PDSW	100	250

The component is installed in a fluid system simulating that to be used in service. A source of contaminated fluid and the necessary pumps or "blow-down" equipment is provided to allow the unit to perform a specified number of operations under simulated service conditions.

A filter containing the finest of the four recommended media is installed upstream of the component under test and the unit is operated for a specified number of cycles under

flowing conditions. The source fluid is contaminated to a relatively high contamination level to assure a meaningful number of particles being exposed to the uni . A level of 30 milligrams of AC Coarse Dust per liter of liquid is recommended. This corresponds to a contamination level approximately 10 times higher than level 350 of Mil-Std-1246, and provides accelerated results. Depending on the total time of operation and flow rate, the reservoir must contain sufficient fluid to complete the run. The test cannot be conducted under recirculating conditions as the returning filtered fluid will constantly dilute the reservoir and reduce the contamination level.

The filter area provided in the upstream filter must be adequate to prevent the filter pressure differential from rising beyond acceptable limits during the run. This information can be obtained from the contaminant tolerance curves contained in Volume I of this report.

If the component successfully completes its life cycle protected by the 10 micron GBR medium, the test is repeated on a second component using the next larger filtration rating medium. This is continued using the coarser grades of media until the rating that causes failure or performance degradation is determined. The coarsest filter medium which will allow the component to perform its design life would then be the optimum medium for use.

3.0 TEST PROGRAM

3.1 General

To test the validity of the test method, a bipropellant valve was furnished by Moog, Inc., East Aurora, New York, for evaluation. The valve was Moog Part Number 54-103, a dual valve equipped with dual inlets and dual shut off features. The detailed test procedure for determining the sensitivity of this valve to particulate contaminant and the results fo this test follow.

3.2 Summary

The Moog valve functioned perfectly under extensive cyclic testing using twenty micron absolute (Glass Bead Rating) and finer filtration. When tested with forty micron absolute and coarser filtration, the valve seat was subject to erosion, thus causing leakage levels higher than specification limits. Following the test series, the pull-in current and flow and pressure-drop checks indicate that the torque motor and armature flexure sleeve assembly were functioning properly. Considering the gross contamination to which this vlave was subjected, it is surprising that the operational characteristics after

the test series were so little degraded.

3.3 Discussion

The fluid flow test system was set up in accordance with the schematic shown in Figure 6 of Appendix I of this section. A tare differential pressure of 4 psid max. was obtained at a flow rate of 3.5 gpm in each valve system using deionized water.

The Moog Bipropellant Valve was installed in the system and the necessary electronic control system was assembled in accordance with Figure 7. The valve was actuated open and flow of deionized water was established to give a net pressure differential of 33 psid across the valve. The flow at this differential setting is 3.8 gpm. Storage tank pressure was set at 110 psi. The tank was then calibrated for delivered volume by emptying into a pre-weighed drum and reweighing. Usable tank volume was 37 gallons. The contaminant weight for addition to a full tank of deionized water was then calculated to be 4.2 grams. This correlates to the established contamination level of 30 milligrams AC Coarse Dust per liter of water.

At the start of each test fun, water flow was reestablished at 3.8 gpm and the valve was pulsed at a rate of 10 cycles per second until the water supply was depleted. This resulted in a variation of cycles between 3100 and 4100 for each test run. At the end of each test run, the large filter screens were removed from the 293 mm diameter filter holders and thoroughly cleaned. The valve was purged through the flow system using GN_2. This was followed by a five minute flush with isopropyl alcohol to remove remaining traces of water. Purging with GN_2 was again performed to evaporate the alcohol leaving the valve completely dry.

The valve was then actuated shut and a leak test was performed with an upstream GN_2 pressure of 290 psig. Leakage was monitored for thirty minutes downstream of each side of the valve using a "captured bubble" method. This leakage was recorded in addition to cumulative valve cycles.

With a 10-micron absolute filter screen in the oxidizer side of the system and a 20-micron absolute filter screen in the fuel side, 20,754 cycles of valve operation were performed with zero leakage across the valve seats. A 40-micron absolute filter screen was then installed in the fuel side of the system and a 100-micron absolute filter screen was installed in the oxidizer side. Using this combination, further test runs were made.

After the first run of 4078 cycles, leakage of 1.5 scc/hour was measured on the fuel

side of the valve and 33.2 scc/hour on the oxidizer side. During the second test run, a marked change occurred in the valve closing sound. The run was terminated after approximately 1700 cycles. Subsequent leakage check showed leakage through both sides of the valve beyond specification limits indicating poppet and/or seat degradation. During all test runs, contaminant was maintained in suspension by constant agitation with GN_2. A transducer was installed in the flow system immediately upstream of the valve to measure the amplitude of the pressure wave caused by the valve closing. This pressure increase was negligible. This may have been due in part to the 1/2" line size and in part to the dampening effect of the large volume of gas entrained in the flow stream.

The overall test set-up is shown in Figure 8 with a detailed view of the flow system adjacent to the valve shown in Figure 9. Results of the leakage tests are shown in Table I.

3.4 Post-Test Evaluation

At the conclusion of testing, the Bipropellant Valve was returned to Moog, Inc. for examination and evaluation. Following are the results of the examination.

Tests were performed in an effort to disclose possible damage to the torque motor, flexure sleeve assembly or the Teflon seats in the flapper buttons, since, damage in these areas could have caused the leakage noted.

1. Drop Out Current & Leakage Test

This test was performed by energizing the coils at 30 vdc and pressurizing both ports to 2^{+2}_{-0} psig. Slowly the voltage was decreased while monitoring the coil current, until a marked decrease in outlet gas flow is noted (outlet ports are vented to a water pan). The current level at this point was recorded as the "Drop Out Current."

Coil current was then decreased to 50^{+2}_{-0} ma. and seat leakage measured from each of the outlet ports.

Results:	Limits
Drop Out Current 58 ma.	≥ 50 ma.
Drop Out Leakage Fuel: 0.1 cc/3 min.	≤ 5 cc/3 min.
Ox: 0.3 cc/3 min.	≤ 5 cc/3 min.

2. Flow and Pressure Drop Test

This test was accomplished by applying 28 vdc to the torque motor and 247 psig to both

TABLE I

LEAKAGE TESTS

Fluid: GN_2

Fluid Pressure: 290 PSIG

Test Type: Captured Bubble

Exposure Time: 30 minutes. (each side)

Test Series A: Oxidizer Side: 325 x 2300 TDDW 10
                       Fuel Side: 2 x 120 x 650 PDSW 20

CYCLES	LEAKAGE scc/hr		CYCLES	LEAKAGE scc/hr	
	Oxid.	Fuel		Oxid	Fuel
0	0	0	14012	0	0
3169	0	0	17169	0	0
6622	0	0	20754	0	0
10588	0	0			

Test Series B: Oxidizer Side: 30 x 160 PDSW 100
                       Fuel Side: 80 x 400 PDSW 40

CYCLES	LEAKAGE scc/hr		CYCLES	LEAKAGE scc/hr	
	Oxid	Fuel		Oxid	Fuel
0	0	0			
4078	33.2	1.5			
5735	>100	>100			

inlet ports. Then by controlling downstream back pressure, fuel and oxidizer flow rates were recorded at several pressure drops. This information was finally plotted and the differential pressures recorded at flow rates of 0.568 lb/sec and 0.707 lb/sec for the fuel and oxidizer ports respectively.

Results		Limits
Fuel	30.6 psid ——	25 - 36 psid
Oxidizer 30.9 psid		25 - 36 psid

3.. Pull In Current Test

The current drawn by the coils when the valve opens was observed. Both ports were pressurized to 247 psig, and coil voltage was increased from zero toward 30 vdc while monitoring coil current.

Results:	Limits
268 ma	$\leq$400 ma.

4. Inspection Test

The manifold was removed and the buttons inspected and photographed. (See Figures 1 - 4).

If the unit had been subjected to a normal environment, test 1 above would have been the more difficult leakage test to pass. However, after viewing the pitted and particle imbedded nature of the buttons, it was decided that an additional leakage check would be made at zero signal to the coils. First, leakage was measured at 7 psig applied to both inlets, and then 290 psig.

@ 7 psig:	Fuel 0.1 cc/12 min.	Oxid. 0.8 cc/12 min.
@ 290 psig:	Fuel 0.3 cc/12 min.	Oxid. 25.8 cc/12 min.

(This test is normally run at 247 psig, the leakage limit for which is $\leq$1 cc/12 min.)

3.5 Conclusions

The pull in current and flow and pressure drop tests indicate that the torque motor and armature flexure sleeve assembly functioned properly. Also, the button travel conformed to specification requirements.

Analyzing the photographs and leakage test results, there is no doubt but that the excessive leakage at the higher pressure level was due to orifice type leak paths which allow more flow the greater the drop across them. Note the erosion (oxidizer button), pitting and radial impressions left by large stringers having laid across the manifold
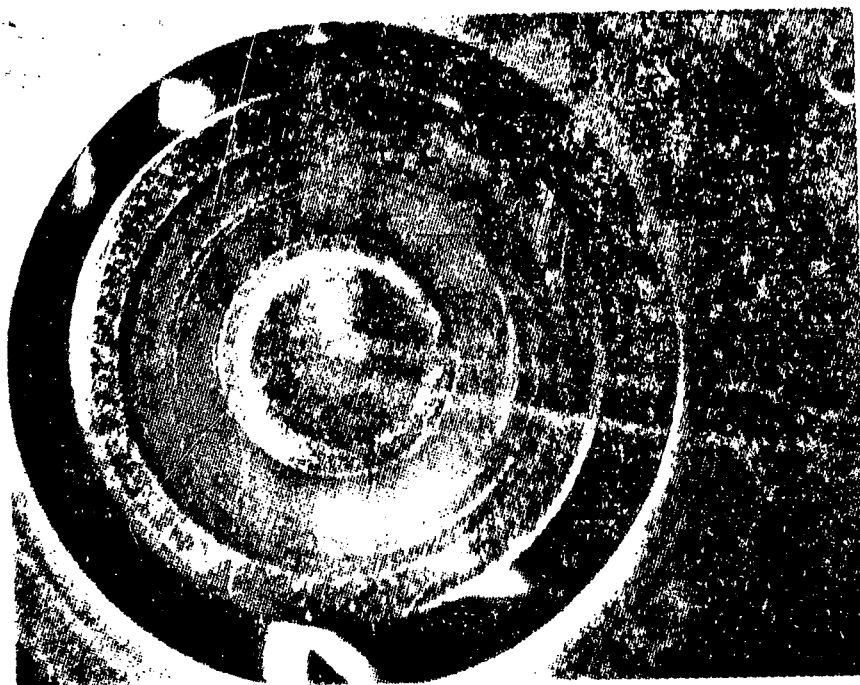
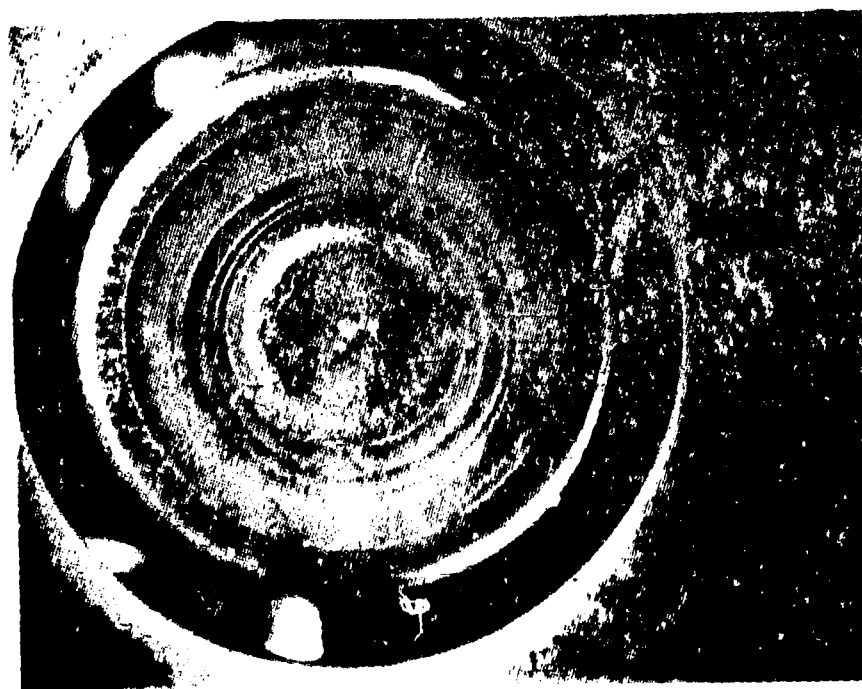FIGURE 1

OXIDIZER BUTTON

(Direct Lighting,   7. 5 X Magnification)

FIGURE 2

FUEL BUTTON

(Direct Lighting,   7. 5 X Magnification)
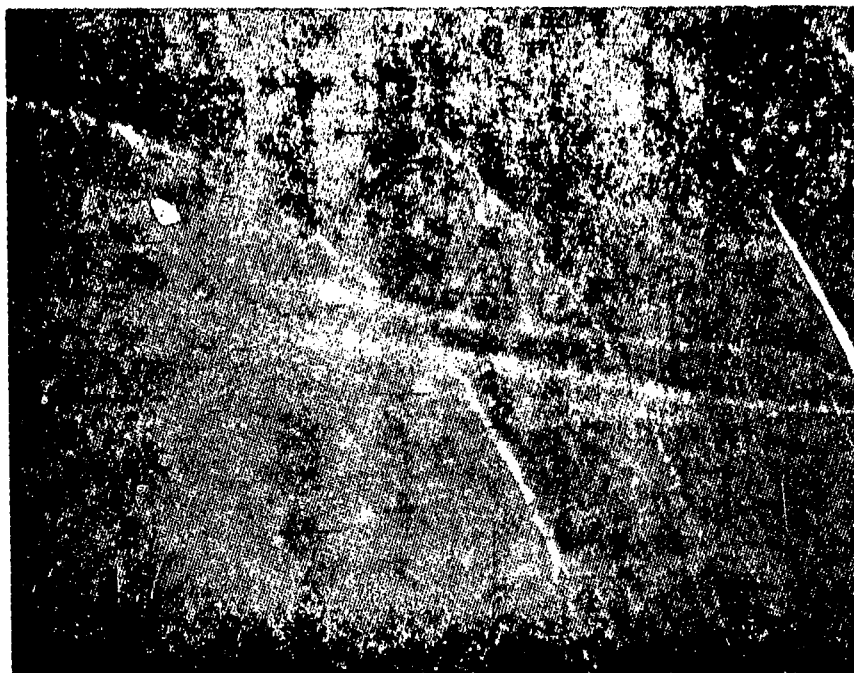
FIGURE 3

OXIDIZER BUTTON

Indirect Lighting,   40X Magnification

FIGURE 4

OXIDIZER BUTTON
Direct Lighting,   40X Magnification
(This is not the same area shown at top of page)

sealing impression. Any or all of the discrepancies could have caused the leakage in question.

Inasmuch as the valve showed performance degradation when the 40 micron GBR filter was installed upstream and no degradation when protected by the 20 micron media, it can be concluded that this particular component is "sensitive" to particles larger than those which will pass through a 20 micron GBR medium. The optimum filter medium to be used for upstream protection would then be the 2 x 120 x 650 PDSW, or the alternate medium, 165 x 1400 TDDW. While the 325 x 2300 TDDW (10 micron GBR) would also provide adequate protection, the pressure drop across this material would be higher and the service life shorter than with either of the recommended media.

TEST PROCEDURE TP-1

SENSITIVITY TEST OF MOOG BIPROPELLANT VALVE

1.0 SCOPE

The purpose of this series of tests is to determine the sensitivity of the Moog Incorporated Bipropellant Valve (P/N 010-50250) to particulate contaminant (AC Coarse Dust) in terms of the maximum micron rating filter required at the valve inlet to prevent malfunction caused by particulate contamination.

2.0 INTRODUCTION

The test consists of a series of simulated operational runs using test fluids precontaminated with AC Coarse Dust to a specific particulate contamination level. The contaminant level will be 30 mg. AC Coarse Dust per liter of test fluid (reference Figure 5). Each run is conducted with inlet filters of specific micron rating. Two runs are performed each with progressively coarser inlet filter media. The following media are used in the order shown:

Oxidizer Valve	Fuel Valve
325 x 2300 TDDW (10μ)	2 x 120 x 650 PDSW (20μ)
30 x 160 PDSW (100μ)	80 x 400 PDSW (40μ)

These media have 10, 20, 40 and 100 micron (μ) glass bead ratings as noted. Periodically and after each run, the valve is tested for leakage and is compared to the pre-test baseline values. Periodic leakage tests will be conducted every 3500 cycles when testing with the 325 x 2300 TDDW and 2 x 120 x 650 PDSW filter media and every 1500 cycles when testing with the 80 x 400 and 30 x 160 PDSW filter media. The tests will be conducted until approximately 21,000 cycles have accumulated with each flow media. The sensitivity of filtration requirements are determined by noting the filter medium at which failure or degradation beyond established limits occurs. The next finer filter medium than that employed in the failure run represents the degree of protection required and, thus, is a measure of the contaminant "Sensitivity" of the valve.

3.0 DESCRIPTION OF TEST ITEM

Part No.:	010-50250
Model No.:	54-103A
Serial No.:	Eng. Eval. #1

The Moog Model 54-103A Bipropellant Valve is used for delta velocity and attitude correction. Each valve incorporates a mechanical linkage to operate the fuel and oxidizer

poppets simultaneously. An electrical solenoid activates the mechanical linkage. The solenoid is energized by an external power source. The poppet seal width is 0.015 inches (400 microns) and the stroke is 0.030 inches.

4.0 TEST PLAN

1. Set up a dual hydraulic flow system as shown on attached schematic, Figure 6.

2. Carefully remove inlet filter from both inlets (oxidizer and fuel).

3. Fluid to be used: deionized water

   Flow Rate: 3.5 GPM (tc rpovide velocity equivalent to .678 lbs/sec N_2O_4 or .424 lbs/sec MMH)

   Pressure Drop: approximately:
   33 psid (oxidizer side)
   33 psid (fuel side)

   Valve Response Rate: approximately (at 247 psig and 24 vdc, reference only)
   .029 second to open
   .007 second to close

   Voltage Range: 24 to 32 vdc

   Supply Pressure: 100_psig approx.

   Line Size: 1/2 inch

   Filtration: Noted on schematic

4. Load system with deionized water that has been pre-filtered through 0.45 micron filters. Assure that flow system is clean. Recirculate through 0.45 micron filters to clean up if required.

5. Install a 325 x 2300 TDDW (10μ) pre-filter in the "oxidizer" valve system and a 2 x 120 x 650 PDSW (20μ) pre-filter in the "fuel" valve system as noted in Figure 6. Check operation of systems without Biprepellant valve installed in flow systems. Electronic instrumentation required is shown in Figure 7.

6. Measure and record the amount of GN_2 leakage through the fuel and oxidizer sides of the valve with 290 psig inlet pressure. The initial leakage should not exceed 5 scc/hr at an inlet pressure of 290 psig.

7. Determine and weigh the amount of contaminant (AC Coarse Dust) required to obtain the desired contamination level (30 mg AC Coarse Dust per liter) of the contaminated water supply systems. Set measured contaminant aside.

8. Install the 010-50250 Bipropellant Vlave Assembly into the flow system. Adjust flow rate and differential pressure to the desired level with the valve actuated to flow position.

9. Add the pre-weighed contaminant in slurry form to the reservoir and maintain uniform suspension of contaminant in the water by agitating the storage reservoir with a constant supply of GN_2.

10 Actuate Bipropellant valve 3500 times while flowing both systems. Permit the con-

taminated water to flow directly into the drain.

11. Close flow valves of both flow systems with the Bipropellant Valve actuated to flow position. Allow system pressure to drop to 0 psig, then close both throttle valves.

12. Flush Bipropellant Valve with pre-filtered isopropanol. Purge dry with GN_2.

13. Measure and record the GN_2 leakage rate through both sides of the valve with 290 psig inlet pressure.

14. Repeat items 4, 8, and 10 through 13 for additional 3500 cycle increments, until 21,000 cycles have been accumulated, or until either the oxidizer or fuel side of the valve fails or exceeds the allowable leakage rate, whichever comes first.

15. Upon completion of the 21,000 cycles noted in item 14, remove the 325 x 2300 TDDW and 2 x 120 x 650 PDSW pre-filters noted in item 5.

16. Install a 30 x 160 PDSW (100μ) pre-filter in the "oxidizer" valve system and a 80 x 400 PDSW (40μ) pre-filter in the fuel valve system as noted in Figure 6. Check operation of system without Bipropellant valve installed in flow system.

17. Repeat items 7 through 13 except - actuate the Bipropellant valve 1500 times while flowing both systems, (instead of 3500 times).

18. Repeat items 8, and 10 through 13 for additional 1500 cycle increments until 21,000 cycles have been accumulated with the new pre-filters, or until either the oxidizer or fuel side of the valve fails or exceeds the allowable leakage rate, whichever comes first.

19. When failure occurs in one side of the valve, continue testing the other side of the valve using the next finer screen to that which was protecting the failed side of the valve. Continue this test to failure. The filter holder protecting the failed side of the valve should contain the 325 x 2300 screen during this final test sequence to prevent further degradation of the failed seat area.

20. Upon completion of all tests, the Bipropellant Valve shall be thoroughly flushed with pre-filtered isopropanol followed by drying, freon flush and baking. The isopropanol and freon shall be filtered through 0.45 micron filters.

FIGURE 5

COMPARATIVE CONTAMINATION LEVELS

FOR

SENSITIVITY TESTS

LOG x LOG SQUARED
6 Cycles x 1-100 Div.
E-599 © 1958 F.W.Cole
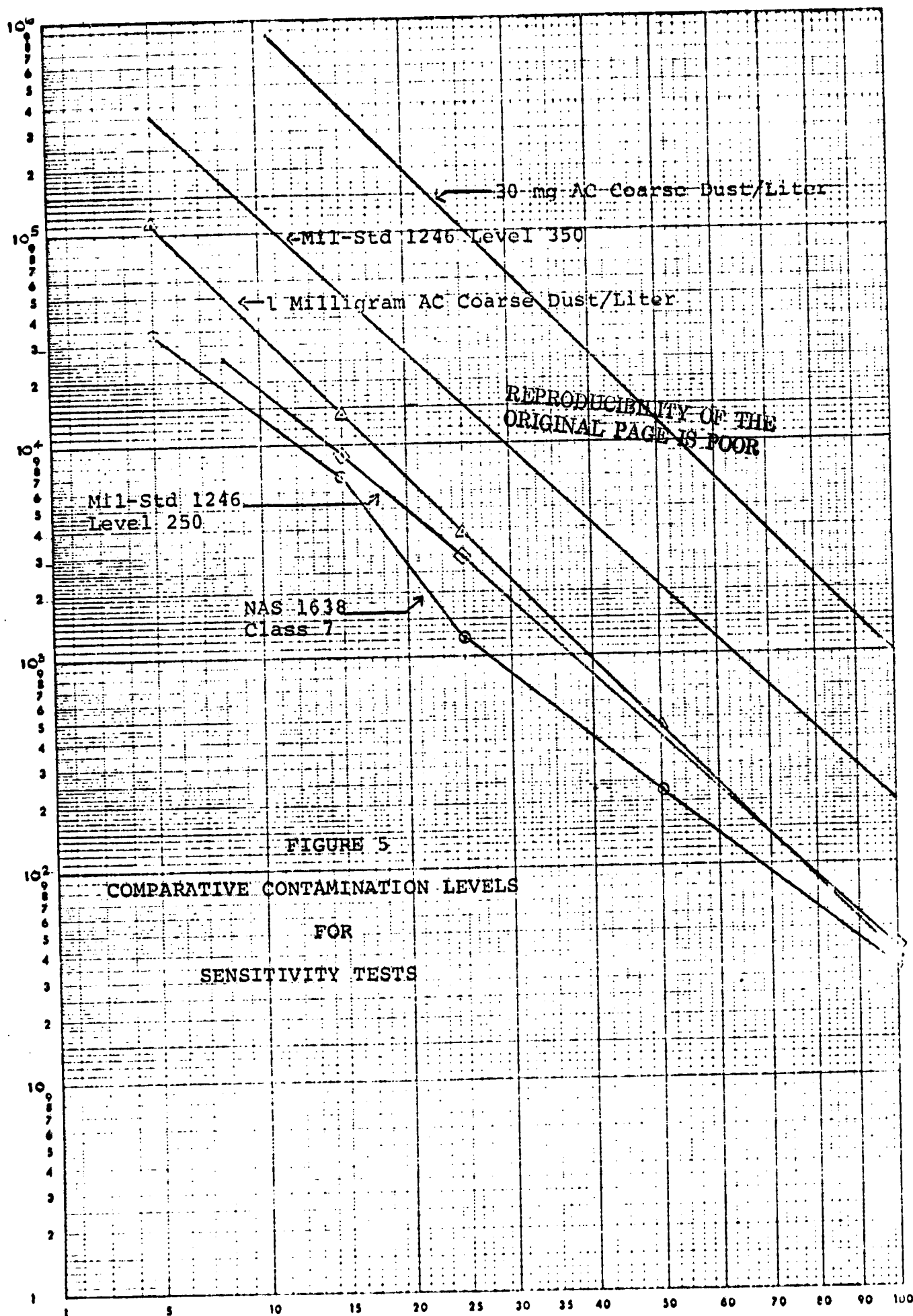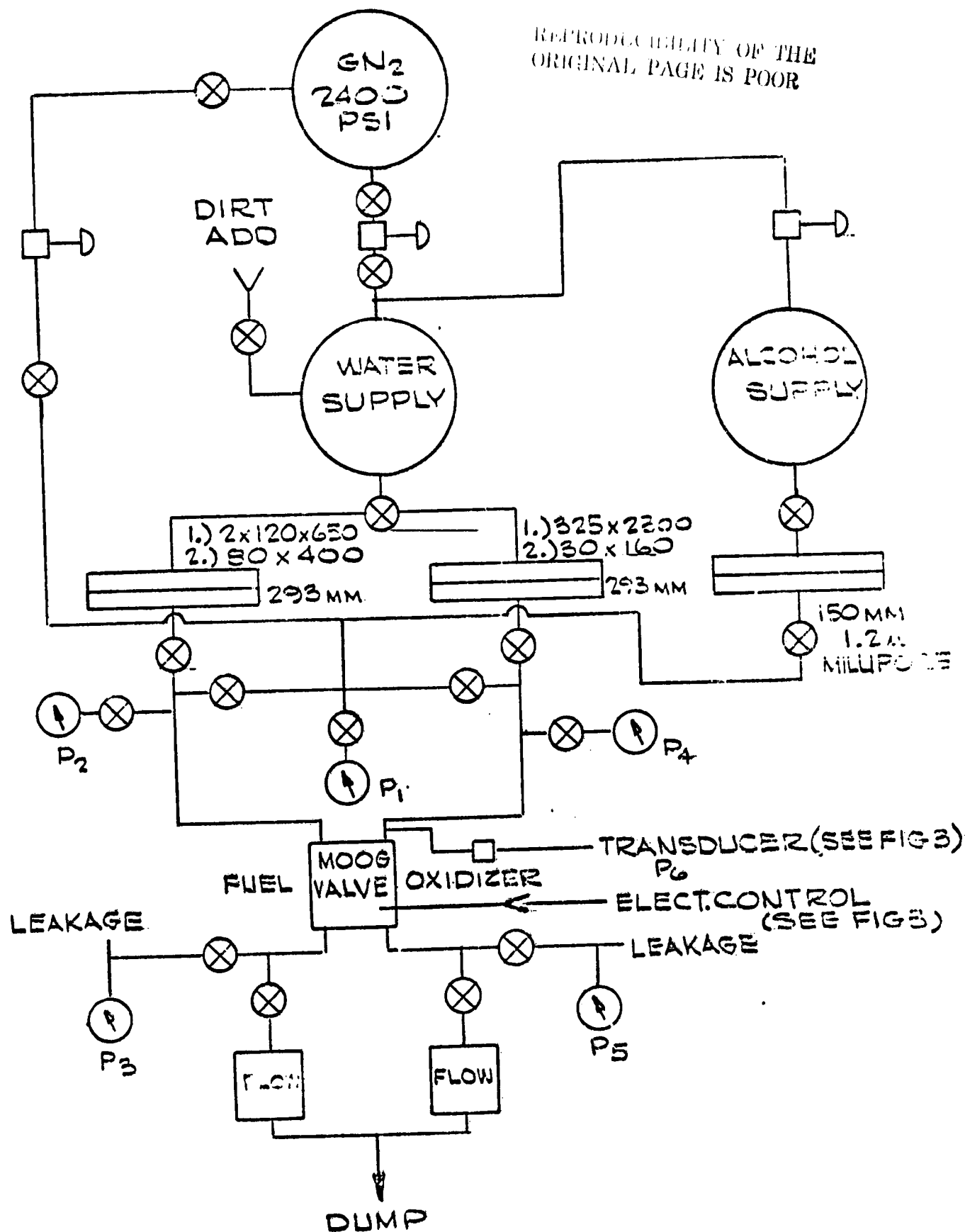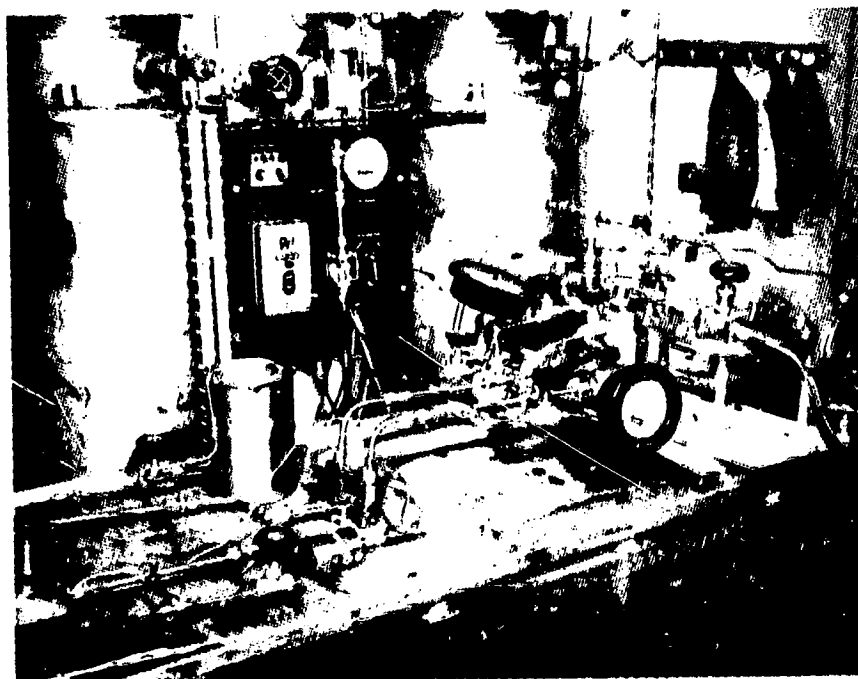
FIGURE 6
TEST SCHEMATIC
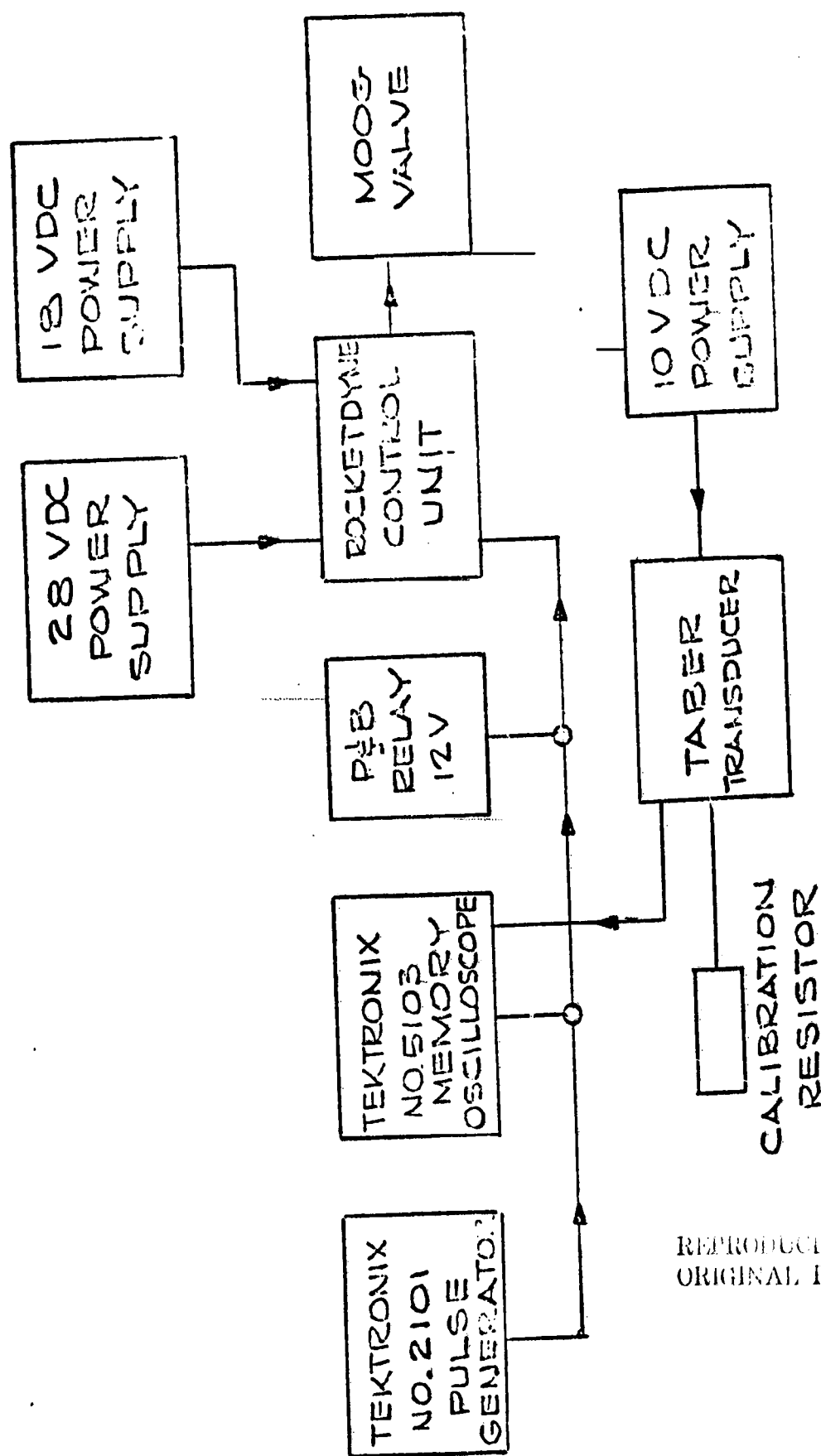
FIGURE 7

ELECTRONIC CONTROLS
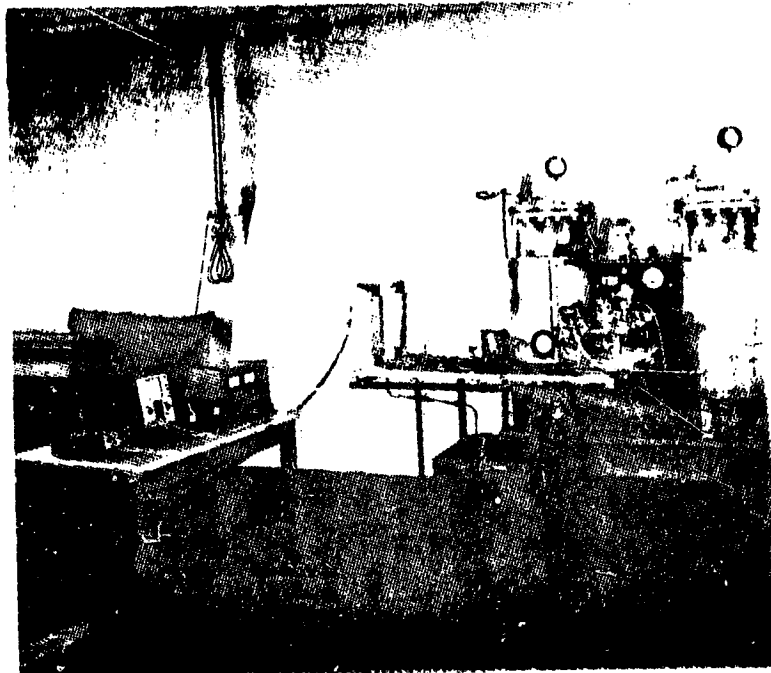
FIGURE 8

TEST SYSTEM

FIGURE 9

FLOW SYSTEM ADJACENT VALVE